Saskatchewan
Agriculture
and Food

ADF

AGRICULTURE DEVELOPMENT FUND

FINAL REPORT

20040462

EXPERIMENTATION WITH ESSENTIAL OIL EXTRACTION FROM THREE NEW SPICE CROPS – CORIANDER, ANISE AND FENUGREEK

Funded by: The Agriculture Development Fund

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Prepared by: Prairie Agricultural Machinery Institute

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1304P

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Development Report

Essential Oil Experimentation

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1. Summary

2007-04787/

This project was designed to help define the operating conditions and equipment settings for optimum essential oil extraction from coriander, fenugreek, and anise.

The project was initially delayed due to lack of availability of test materials and some unexpected results with prior essential oil extraction from screenings of caraway and short sample runs with anise and coriander. Buyers of the oil identified minor quality problems, which may need to be rectified by adjusting either operation parameters or by prior cleaning of raw material. Detailed laboratory testing has been approved as a project change by ADF as a prior step. Two rounds of testing with some attempts at oil purification have now been carried out.

The testing by POS has produced sample results that parallel those provided by oil buyers. Detailed preliminary analysis to determine the nature of contaminants and the potential for their removal has been completed. A request for a further extension was made. This allowed some minor filtering and settling routines to be employed to see if it improved oil quality. Carrying out the procedures caused little visual change to the oil samples. The samples were subjected to re-testing at POS with the same test procedures as initially. While some changes in constituents were made, none of them would be particularly beneficial. Changing the sampling procedure for anise oil brought results within tolerance.

2. Introduction

PAMI has been using the large MRF designed counter current steam extractor to remove essential oil from dill and caraway screenings on a contract basis for spice seed processors. The processing requirements are understood, and extraction rates have generally been excellent with oil quality satisfactory. Detailed methods to properly extract the oil from coriander, anise, and fenugreek are unknown, and some trial runs were unsuccessful. Pre-processing may be required, and operating parameters may change. This project was designed to help define the conditions needed for extraction from coriander, fenugreek, and anise.

A secondary issue arose when processing screenings of coriander and anise produced essential oil that buyers considered to be off grade, and as such, of low value. An attempt to resolve this issue is now being made as part of this project prior to proceeding further.

Preliminary tests showed that the buyer's tests were accurate and indicated potential sources of contamination. Some contaminant sources could be eliminated by pre-processing methods, but the potential also exists to purify the oil already processed.

Several simple cleaning processes were tried. These were intended to improve oil quality to allow sale of the product and thus improve the economics of oil extraction from screenings. The effectiveness of these cleaning processes was tested and is included in this report.

3. Test Procedure

The last batch of screenings processed for G.H. Schweitzer Enterprises did not meet one client's requirements. The following problems (**Table 1**) were identified with the corresponding consequence to oil sales:

Table 1. Processing Problems and Corresponding Consequence to Oil Sales

Caraway Oil	
Refractive Index (RI)	High (1.49 vs 1.48 max) - Saleable but must be blended
Specific Gravity	High (0.928 vs 0.91 max)
Optical Rotation	Low (65.8 to 68 vs 70 min)
Carvacrol Content	High (66 to 70%) – OK
Dill Oil	
Specific Gravity	High (0.918 vs 0.919) – Saleable
Optical Rotation	Low (66.3 to 68.7 vs 70 min)
Carvone Content	High (63%) – OK
Coriander Oil	
Specific Gravity	Good
Refractive Index	Good – Questioned Purity
Optical Rotation	Good – Ratio of Peaks
Linalool Content	51.29% - Low (Different from Cilantro and Coriander Seed)
GC - MS	Ratio of Peaks - Extra peak found on GC-MS analysis
Anise Oil	
Specific Gravity	Low (0.997 vs 0.978 min)
Refractive Index	1.549 vs 1.553 min
Optical Rotation	1.4 vs 1.0 max
Odour	Not Right
85 to 90% Anethole	GC not typical of Star Anise or Pimpinella

The buyer's main concerns are with odour, extra peaks, and marginal characteristics. Adulteration with other oils is always a possibility, and some similar oils have toxic characteristics. As such, precise conformance to a usual characteristic is important. The oils are all a combination of various components. Changing a relative quantity of a component changes specific gravity, optical rotation, and refractive index. Inpurities, such as dust, moisture, or heavier components, can also affect these.

With caraway and dill, the developed and optimized procedure increases carvone content and reduces relative limonene content by maintaining a higher condenser temperature to bypass some limonene. This may have caused the lower than expected optical rotation and higher specific gravity, as carvone is a heavier component. In any case, the buyer did not reject the oil.

The coriander oil has a different ratio of peaks than expected, and the buyer questions whether it is a blend of cilantro and coriander seed oil. This is likely as screenings may contain leaf or stem material, but the "extra" peaks are possibly from a contaminant.

The anise oil has a high, anethole content, but the GC is untypical of what is expected, and odour is not right. In this case, the specific gravity is low, so there may be lighter components (limonene) that their other suppliers bypass by using higher condenser temperatures. It may also have a different odour due to contamination.

This part of the project was planned to solve the problems with quality of processed essential oil from caraway, coriander, and anise. Initial testing was carried out at POS to answer the following questions:

1) Is the buyer's analysis correct?
2) Would laboratory extraction arrive at the same results in terms of oil quality as that produced by PAMI equipment?
3) Could the source of "off-grade" be determined? Is it due to processing method, basic contamination of raw material supply, or contamination due to inadequate clean out between runs?

Approximately 50 ml samples from each of three anise oil barrels, one caraway barrel, and one coriander barrel were extracted using a glass tube from approximately the middle of each oil volume. These, along with samples of approximately 1 kg of un-extracted raw material as provided by Gary Schweitzer from bin samples, were sent to POS for detailed testing.

Oil samples both provided and extracted by POS were analyzed for refractive index and specific gravity and compared to buyer's preferred target.

GC-MS analysis of samples was carried out, and test results were compared to results of the buyer's testing.

4. Test Results

4.1 Comparison of Buyer's Oil Lab Results to POS Lab Results

Table 2 shows a comparison of POS test results to buyer's test and to preferred results for refractive index and specific gravity. Angle of rotation testing was not carried out, as the equipment was not readily available at POS.

Table 2. Comparison of POS and Buyer Test Results.

POS Lab				
Sample	**Refractive Index**	**Preferred Result**	**Specific Gravity**	**Preferred Result**
Anise 1	1.553	1.553 Min	0.985	0.978 Min
Anise 3	1.549	1.553 Min	0.984	0.978 Min
Anise 5	1.553	1.553 Min	0.989	0.978 Min
Caraway	1.49	1.48 Max	0.929	0.91 Max
Coriander	1.462	OK	0.870	OK

Buyer's Lab				
Sample	**Refractive Index**	**Preferred Result**	**Specific Gravity**	**Preferred Result**
Anise (Lot 16)	1.552 (Pass)	1.553	0.989 (Pass)	0.978 (Min)
Anise (Lot 17)	1.549 (Low)	1.553	0.977 (Low)	0.978 (Min)
Caraway (Lots 1 to 14 Average)	1.490	1.48 (Max)	0.921 to 0.930	0.91 (Max) (High Carvacol)
Coriander (Lot 15)	1.463	Not Available	0.869 Meets requirement but has unknown ingredients– bad odour	Not Available

4.2 Analysis of Comparison of Lab Results on Extracted Oils

A review of tests generally shows that the results of buyer testing are similar to the POS laboratory tests. The source of the preferred results standard (**Appendix III**) was provided by the buyer. The buyer questioned whether there was a mixture of coriander and cilantro in the coriander material.

The failure criteria were:

1) Coriander – Bad odour and possible mixture of coriander and cilantro oils. Extra peaks were found on the GC-MS result.
2) Anise – Lot 16 marginally passed; Lot 17 did not. It was noted that odour was a little off and some confusion as to the GC not matching either pimpinella or star anise exactly.
3) Caraway – Specific gravity was too high consistently (due to higher carvone preferred by some buyers), purity was excellent, high on refractive index, and low on optical rotation.

4.3 Comparison of POS Extracted Oil to PAMI Extracted Oil

4.3.1 Comparison of Caraway Sample 1 to Extracted Caraway Bin 121

This sample shows that the POS Pilot Plant extraction has a higher limonene value (42.65 vs. 30.04%) than the PAMI sample (**Table 3**). This was due to the intentional optimization of process at PAMI to increase the carvone value. It is noted that the POS method extracts one heavier compound anethole and one lighter compound than found in the PAMI sample. These may be lacking in the buyer sample due to the difference in procedure at PAMI or a settling in the barrels at PAMI prior to sampling.

Table 3. POS Pilot Plant Sample vs. PAMI sample.

	Refractive Index	***Specific Gravity***
PAMI Caraway Sample 1	1.4901	0.9285
Extracted Bin 121 Screenings	1.4876	0.9261
Buyer Standard	1.48 (max)	0.91 (max)

The sample results are basically identical. The differences are probably due to the very high carvone reading. Carvone is a heavier compound, so the higher percentage increases specific gravity. The buyer is actually getting a preferred quality, not a poorer quality, but some buyers will still be concerned because it is not identical to prior purchases.

No obvious repair solution presents itself. It may be a feedstock problem though increasing the limonene content by changing our condenser temperature could reduce specific gravity very slightly and change the analysis.

The results show that the samples contained only limonene and carvone, as they should. The POS extraction process retained a lighter compound and anethole, which no doubt was bypassed in our system by using a higher condenser temperature. The fact anethole is there suggests there is some anise contamination in the screenings, but the MRF system removes it.

4.3.2 Detailed Anise Sample Comparison to Extracted Anise

Three anise oil samples were provided to POS for analysis. We sampled three barrels, which provided Samples 1, 3, and 5. Extracted screenings samples were from Bin 23 and a non-specified origin sample provided by G.H. Schweitzer Enterprises. It is noted that anise Sample 1 and the unprocessed sample provided similar analysis, and Bin 23 and Sample 3 were similar. Sample 5 is somewhat different as expected. It was taken from a settled-out barrel. In the upper portion of the barrel, the heavier compound, a-gurjunene, would be excluded. Comparison with the buyer's results is shown in **Table 4**.

Table 4. Comparison with Buyer's Results

	Refractive Index	Specific Gravity	% Anethole
Anise 1	1.5531	0.9850	91.69
Anise 3	1.5492	0.9804	85.59
Anise 5	1.5529	0.9897	92.07
Buyer Lot 17	1.549	0.977	89.57
Buyer Lot 16	1.552	0.989	85.36
Extracted Unprocessed	1.5480	0.9913	88.32
Extracted Bin 23	1.5464	0.9867	87.26
Buyer Standard	1.553 (min)	0.978 (min)	75 to 90

These results suggest that the PAMI extraction worked correctly but may have bypassed some limonene and has included some a-gurjunene. The oil is expected to contain estragole, anise ketone, and beta caryophyllene. The main issue appears to have arisen due to the poor test result of Lot 17, which had low specific gravity and slightly lower RI. It may have been a poor sample. Anethole numbers are excellent, but Sample 3 and Buyer Sample 17 were probably top-of-barrel samples after settling. The odour issue may be due to contamination with weed seeds. It is noted that the samples contains carvone, which may not be ideal and could have easily been provided from caraway, dill,

or coriander contamination of feedstock, or in carry over contamination from a previous run. The similarity between the POS extracted and MRF equipment extracted sample suggests the problem is most likely in the raw material.

Upon examination, it was noted that the standard procedure for anise oil analysis is to warm the oil and mix it before sampling. This was not done prior to sampling, so a retest with new samples may result in passing the refractive index and specific gravity tests.

4.3.3 Coriander Samples – Detailed Comparison of Extracted Oils

One sample of extracted coriander oil - Sample 1 - was analyzed and compared to extracted samples from two sources. The buyer's concerns are an extra "peak" which could be a contaminant, and the color and odour was thought to not be good.

An examination of the buyer analysis shows the extra peak at 22.495 signal one time. A comparison of the peak position to linalool peak and a comparison to sequence both identify the unidentified peak as being either carvone or anethole. The area of 7% is consistent with it being either carvone or anethole. The carvone percentage varies from 4.92 to 15.83%, and anethole varies from 1.32 to 17.22%.

This obviously could indicate contamination with anise, as anise contains both anethole and carvone. The carvone content in anise is usually low, so it appears there could be cross contamination in process from prior runs of anise in the equipment or by raw material contamination.

The anise was processed by PAMI before the coriander. The extractor and condenser were cleaned, but it may not have been adequate. The heavier anise oil may have adhered to tubes in the condenser. While it was steamed and flushed out, it should likely have been heated to 100°C (a boil out) to ensure all traces of anise oil were eliminated. It may be possible to remove anise contamination from the coriander oil by heating and recondensing. The anise oil is heavier than the coriander oil and should evaporate last. The fact that the POS extracted material has the same contamination confirms that the raw material is contaminated.

4.4 Preliminary Conclusions from First Testing Round

The preliminary conclusions were that:

1) The buyer's analysis was correct.
2) The PAMI equipment and laboratory extraction arrived at similar results, so the oil quality was likely contaminated by mixed screenings containing off-type seeds.
3) The anise oil may have been sampled incorrectly.
4) The coriander had an extra spike indicating contamination. While a potential cause could have been contamination during a change over at PAMI from anise, this is

unlikely as the POS test results from the raw coriander screenings also yielded the same extra spike. This indicates that the screenings may contain a contaminating component and that it may be anise. A visual examination of samples did not confirm this.

4.5 Filtering and Chilling Techniques

The oil was run through paper filters after warming to improve flow. Caraway and dill oil were warmed to 20°C, while anise and coriander oil were warmed to 45°C to obtain adequate flow. The filtered oils were then chilled and after precipitation of solids at the lower temperature, the liquid portion was sampled again.

4.5.1 Sample Descriptions

A second set of testing was conducted to employ filtering and temperature change to try to separate contaminants. Sample tested were:

1) Caraway – filtered only.
2) Dill – filtered only.
3) Caraway – chilled to 8°C and filtered.
4) Dill – chilled to 8°C and filtered.
5) Chilled Caraway – sampled filtered oil from below precipitates at top of column.
6) Chilled Dill – filtered oil from below precipitates. (Note – precipitates like icicles are present at -10°C below surface of the oil.)
7) Coriander Oil – filtered only.
8) Coriander – as filtered at 8°C. (No visible precipitation—slow to filter—no gelling of oil).
9) Coriander – filtered at 0.5°C to -2°C. (Note – a layer precipitated out on cooling.)
10) Coriander – at -6°C, no visible precipitate—no gelling of oil.
11) Coriander oil – heat to 60°C—filtered.
12) Anise – barrel 1 – filtered after heating and stirring.
13) Anise – barrel 2 – filtered after heating and stirring.
14) Anise – barrel 16 – settled – filtered.

These samples were sent to POS for further testing. The following are the effects of simple filtering and reduced temperature filtering techniques to attempt to purify the extracted essential oils.

4.5.2 Caraway and Dill Oil Results

These oils as originally tested, were considered to be saleable. The properties were only slightly off standards, but carvone contents were high.

They were filtered to produce first samples, chilled after filtering to 8°C and re-sampled and then chilled to -10°C, and the liquid sampled again.

There was no significant change in Specific Gravity, Refractive Index, and GC Analysis with the first two tests on dill oil. The -10°C chill did condense out most of the limonene, resulting in 97% pure carvone.

With the caraway oil, the percentage of carvone increased with both chilled samples. The benefit of this improvement would not likely be of financial benefit as both dill and caraway had initial carvone requirements exceeding the requirements of buyers. Improving concentration would reduce value unless price premium on higher concentrations were available.

4.5.3 Anise Results

Anise oil in barrels 16 and 17 is settled out into two constituents. As such, it was warmed to 45°C and stirred to mix the components together. One sample was taken from barrel 16 of the separated upper liquid component called the "Anise Initial" sample. The main mixed material from both barrels was then chilled to -25°C and to fridge temperature. Both crystallized completely and were not re-sampled.

The proper warming and mixing sampling routine resulted in an anise analysis that now meets buyer requirements for Refractive Index and Specific Gravity. Inspection of the GC results of barrel 17 shows it contains terpine, which is not in barrel 16. Terpine is also the "contaminant" in coriander. So it may have been cross-contaminated during the process when the coriander out-feed-auger was run for a few minutes in error instead of the anise out-feed-auger. When it was realized, the feed auger was stopped and the surge bin and augers were cleaned out. It is unlikely any of that material, or a significant amount, reached the processor.

An examination of the "anise unprocessed" samples showed a lot of stem material, some coriander hulls, and a large variety of other material. Many of these ingredients are in the coriander.

Our conclusion is that the first anise material run (barrel 16) was much cleaner and the source of the "terpine" is somehow related to other debris contamination in the screenings, perhaps from cleaning coriander or caraway.

4.5.4 Coriander Results

Samples from barrel 15 were filtered first and then samples of the filtered material were re-filtered at 8°C, 0.5°C, -6°C, and then filtered again after reheating to 60°C. Very small amounts of precipitate came out during chilling.

Physical properties were adequate from initial tests. A review of Internet sources shows coriander oil to contain linalool as a primary ingredient with decaldehyde, borneol, geronial carvone, and anethole in lesser quantity. Two components in the oil, terpine and limonene, are not supposed to be there and the anethole content is too low.

This probably has resulted from the inclusion of contaminant stem or seed material and a failure to extract oil from the seeds. The seedpods prevented the steam from removing the seed oil. Rolling, hammermilling, or some other pod breaking method is needed for this process to work with coriander.

GC properties did vary slightly with each test, but the primary contaminants "B terpine" and "limonene" did not. The initial readings in the 8.8% for terpine and 6% for limonene were also still present. Other ingredients are expected in these oils, but not at these levels, and filtering and chilling methods were of no benefit to remove this material. Basically, cleaner screens are needed.

5. Final Conclusions

1) Processing screenings to remove dill and caraway oil seems to work well with little contamination, but this may be due to the screenings being primarily small and broken seed.
2) Anise processing is subject to cross contamination from other seed types and stem material and it may be worthwhile to do sample pre-cleaning.
3) Coriander processing requires a new technique of extraction and/or cleaner screenings to be successful.
4) Simple heating, cooling, and filtering techniques did not improve sample quality, but anise oil must be heated and stirred before sampling.

7. ICAR Data Entry

The associated ICAR number for this project is #33331997.

APPENDIX I

Appendix I

POS Testing Results – Round 1

Processing Solutions

RECEIVED
JUL - 4 2005

PILOT PLANT CORP.

June 29, 2005

Mr. Phil Leduc
PAMI
PO Box 1150
Highway 5 West
Humboldt, SK
S0K 2A0

Dear Phil:

Please find copy of the essential oil data.

If you should have any questions, please don ot hesitate to call.

Sincerely,

C. Wiebe

(for) Rick Green, M.Sc.
Senior Scientist

/cw
Enclosure

PDS Pilot Plant Corp., 116 Veterinary Road, Saskatoon, Saskatchewan, CANADA S7N 2R4
Telephone (306) 978-2800 · FAX (306) 975-3766 · Email: pds@pds.ca · http://www.pds.ca

processing Solutions

PILOT PLANT CORP.

Refractive Index and Specific Gravity of Essential Oil Samples

Project 05-802

Sample	Refractive Index	Specific Gravity
Anise sample 1	1.5531 ± 0.0005*	0.9850 ± 0.0010
Anise sample 3	1.5492 ± 0.0004	0.9804 ± 0.0013
Anise sample 5	1.5529 ± 0.0005	0.9897 ± 0.0026
Caraway sample 1	1.4901 ± 0.0001	0.9285 ± 0.0013
Coriander sample 1	1.4619 ± 0.0003	0.8702 ± 0.0029
Extracted Anise unprocessed**	1.5480 ± 0.0001	0.9913 ± 0.0006
Extracted Anise bin 23 screenings	1.5464 ± 0.0001	0.9867 ± 0.0016
Extracted Caraway bin 121 screenings	1.4876 ± 0.0001	0.9261 ± 0.0017
Extracted Coriander off-grade bin 3	1.4674 ± 0.0001	0.8879 + 0.0018
Extracted Coriander screenings unprocessed 040614-003	1.4753 ± 0.0004	0.8928 ± 0.0040

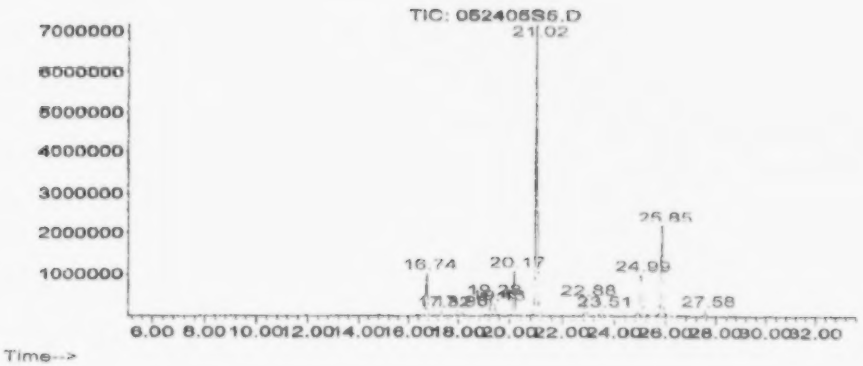
* Sample standard deviation of three replicates.

** Essential oil was extracted from the seed samples by AOAC method 962.17 (using a Clevenger apparatus).

POS Pilot Plant Corp., 118 Veterinary Road, Saskatoon, Saskatchewan, CANADA S7N 2R4
Telephone (306) 978-2800 · FAX (306) 975-3766 · Email: pos@pos.ca · http://www.pos.ca

PILOT PLANT CORP.

GC-MS Analysis



TIC: 052405S5.D

EXTRACTED CORIANDER SCREENINGS UNPROSESSED 040614-003

Peak#	Ret Time	Area	% Area	Compound
1	16.741	43265786	6.38	α-pinene
2	17.324	4968435	0.73	camphene
3	17.975	5322483	0.78	β-pinene
4	19.275	16868905	2.49	ortho-cymene
5	19.431	10753905	1.59	limonene
6	20.172	45191766	6.67	terpinene
7	21.020	352241805	51.95	linalool
8	22.882	18323184	2.70	camphor
9	23.508	5814890	0.86	decanal
10	24.992	52017994	7.67	carvone
11	25.845	116785853	17.22	anethole
12	27.576	6477432	0.96	nerol acetate

processing Solutions

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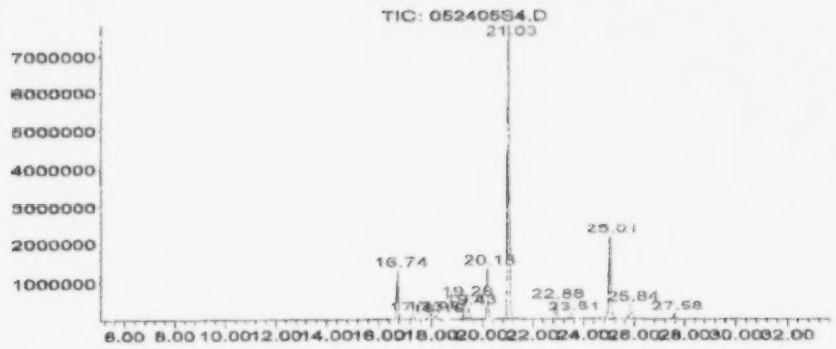
GC-MS Analysis



Coriander off-grade
TIC: 052405S4.D

EXTRACTED CORIANDER OFF GRADE BIN 3

Peak#	Ret Time	Area	% Area	Compound
1	16.744	54854700	7.52	α-pinene
2	17.326	6707635	0.92	camphene
3	17.978	6636065	0.91	β-pinene
4	18.161	4295889	0.59	2-(n-pentyl)furan
5	19.277	22792159	3.12	ortho-cymene
6	19.434	13139277	1.80	limonene
7	20.175	56462232	7.74	terpinene
8	21.026	391746170	53.69	linalool
9	22.884	22109473	3.03	camphor
10	23.511	6487471	0.89	decanal
11	25.005	115483749	15.83	carvone
12	25.836	21471222	2.94	anethole
13	27.578	7409779	1.02	nerol acetate

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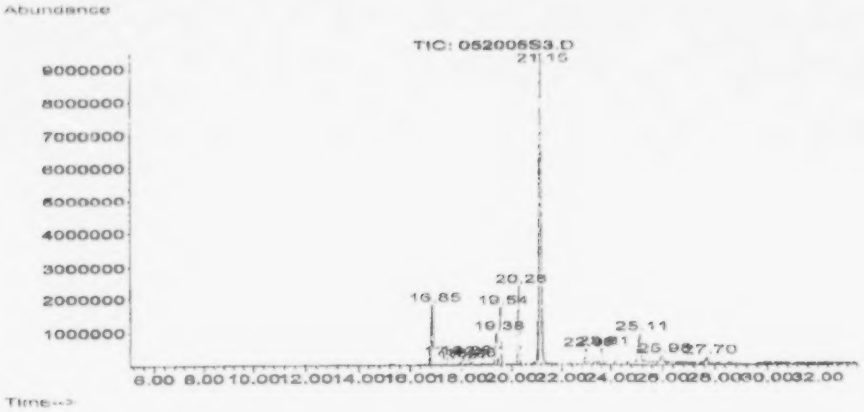
GC-MS Analysis



Coriander

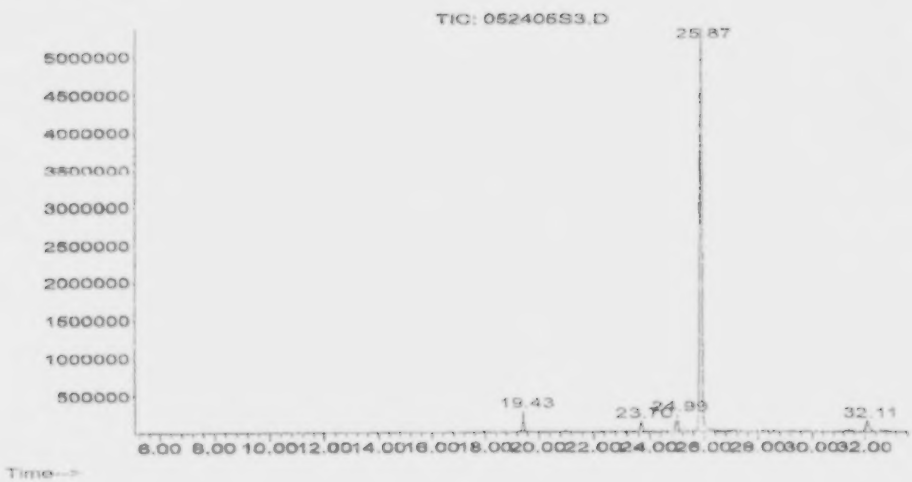
TIC: 052005S3.D
CORIANDER SAMPLE 1

Peak#	Ret Time	Area	% Area	Compound
1	16.848	78153912	7.80	α-pinene
2	17.431	9891243	0.99	camphene
3	17.967	3871297	0.39	β-thujene
4	18.077	9440550	0.94	β-pinene
5	18.264	5985808	0.60	2-(n-pentyl)furan
6	19.383	39619614	3.96	ortho-cymene
7	19.543	69648860	6.95	limonene
8	20.282	102184880	10.20	terpinene
9	21.147	573712359	57.27	linalool
10	22.994	19051446	1.90	camphor
11	23.615	18845646	1.88	decanal
12	25.110	49268942	4.92	carvone
13	25.976	13261346	1.32	anethole
14	27.702	8811639	0.88	nerol acetate

Processing Solutions

PILOT PLANT CORP.

GC-MS Analysis



TIC: 052405S3.D

EXTRACTED ANISE UNPROCESSED

Peak#	Ret Time	Area	% Area	Compound
1	19.431	12068668	3.40	limonene
2	23.696	6722857	1.89	methyl chavicol
3	24.989	11734652	3.30	carvone
4	25.867	313795561	88.32	anethole
5	32.110	10972393	3.09	α-gurjunene

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PILOT PLANT CORP.

GC-MS Analysis

TIC: 052005S5.D
ANISE SAMPLE 1

Peak#	Ret Time	Area	% Area	Compound
1	19.547	9354932	2.10	limonene
2	23.814	9615757	2.16	methyl chavicol
3	25.118	9991406	2.24	carvone
4	26.009	408858198	91.69	anethoic
5	32.295	8077787	1.81	α-gurjunene

POS Pilot Plant Corp., 118 Veterinary Road, Saskatoon, Saskatchewan, CANADA S7N 2R4
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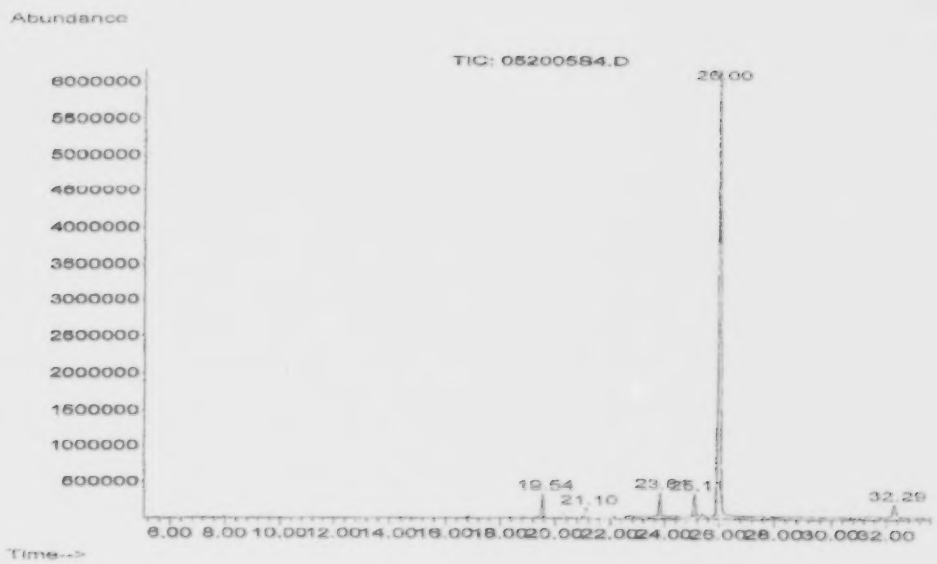
PILOT PLANT CORP.

GC-MS Analysis



TIC: 052005S4.D

ANISE SAMPLE 3

Peak#	Ret Time	Area	% Area	Compound
1	19.543	14050613	3.11	limonene
2	21.100	6648615	1.47	linalool
3	23.811	14884108	3.29	methyl chavicol
4	25.115	16042630	3.55	carvone
5	26.004	386871962	85.59	anethole
6	32.290	13510555	2.99	α-gurjunene

PILOT PLANT CORP.

GC-MS Analysis

TIC: 060305S1.D
ANISE SAMPLE 5

Peak#	Ret Time	Area	% Area	Compound
1	19.549	7297402	3.30	limonene
2	23.811	5310901	2.40	methyl chavicol
3	25.114	4937449	2.23	carvone
4	25.983	203633441	92.07	anethole

POS Pilot Plant Corp., 116 Veterinary Road, Saskatoon, Saskatchewan, CANADA S7N 2R4
Telephone (306) 978-2800 · FAX (306) 975-3766 · Email: pos@pos.ca · http://www.pos.ca

Processing Solutions

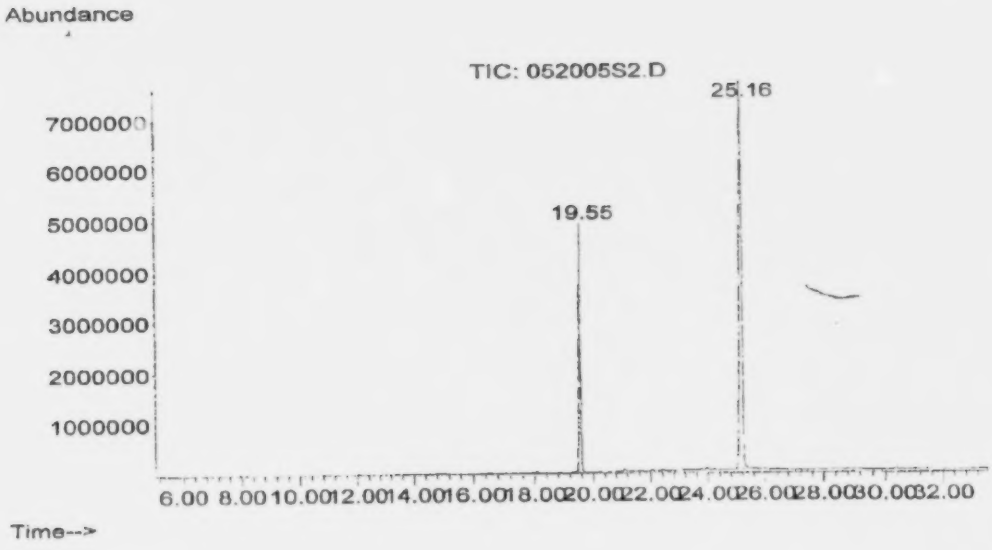
PILOT PLANT CORP.

GC-MS Analysis

TIC: 052405S2.D
EXTRACTED ANISE BIN 23 SCREENINGS

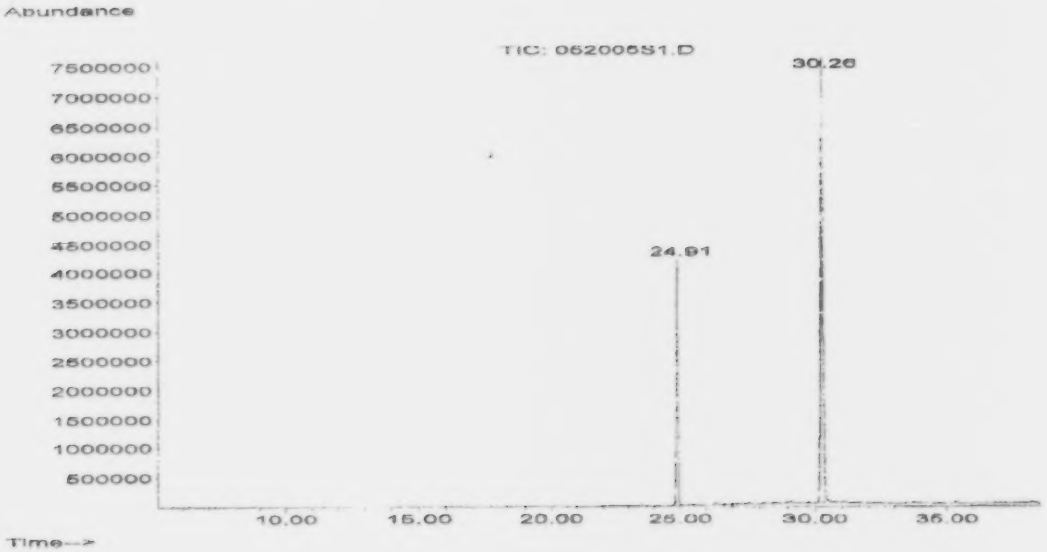
Peak#	Ret Time	Area	% Area	Compound Identification by MS
1	19.427	12738403	3.67	limonene
2	20.985	4781038	1.38	linalool
3	23.691	8332552	2.40	methyl chavicol
4	24.984	9608224	2.77	carvone
5	25.862	302920440	87.26	anethole
6	32.106	8762971	2.52	α-gurjunene

POS Pilot Plant Corp., 116 Veterinary Road, Saskatoon, Saskatchewan, CANADA S7N 2R4
Telephone (306) 978-2800 · FAX (306) 975-3766 · Email: pos@pos.ca · http://www.pos.ca



Caraway 2 ul 65 C
TIC: 052005S2.D
CARAWAY SAMPLE 1

Peak#	Ret Time	Area	% Area	Compound
1	19.547	210884692	30.04	limonene
2	25.155	491201187	69.96	carvone



Caraway 2 ul

TIC: 052405S1.D

EXTRACTED CARAWAY BIN 121 SCREENINGS

Peak#	Ret Time	Area	% Area	Compound
1	5.141	10483973	1.64	2,6-dimethylocta-2,7-dien-6-ol
2	19.455	271995607	42.65	limonene
3	25.029	345318848	54.15	carvone
4	25.833	9926520	1.56	anethole

APPENDIX II

Appendix II

Buyer's Testing Results and Comments

Q/C SAMPLE ANALYSIS REPORT

14-Jul-04

Extra pendo

SE: S-30123

PRODUCT: CORIANDER OIL

SUPPLIER: SCHWEITZER ENTERPRISES

LOT#: LOT 15

PO#:

CONTRACT#:

PRIORITY: RUN EACH DRUM INDIVIDUALLY GIVE RESULTS TO GJ

DATE: 7/12/200

PURITY: 51.29% Linalool LOW

REFRACTIVE INDEX: 1.463 ✓

SPECIFIC GRAVITY: 0.869 ✓

OPTICAL ROTATION: +12.8 ✓ BAD

ODOR EVALUATION:

COLOR/COMMENTS: Pale yellow liquid ✓

RECOMMENDATIONS:

FINAL APPROVAL:

mixture of leaf + seed ??

Injection Date : 7/12/2004 11:38:17 PM Seq. Line : 15
Sample Name : CORIANDER Location : Vial 15
Acq. Operator : JOHN Inj : 1
Inj Volume : 0.1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\071204I.S
Method : C:\HPCHEM\1\METHODS\METHDTDA.M
Last changed : 6/25/2004 1:59:30 PM by JOHN

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	3.735	BB	0.0248	5275.90325	3280.64893	0.11251
2	4.963	BB	0.0353	2.84056e5	1.26053e5	6.05775
3	5.610	BP	0.0391	3.17479e4	1.27427e4	0.67705
4	5.760	BV	0.0402	1495.92178	579.67590	0.03189
5	5.835	VB	0.0346	1766.26535	775.04083	0.03767
6	6.311	BB	0.0423	2.40663e4	8977.70996	0.51323
7	6.485	BP	0.0406	1.51690e4	5787.12500	0.32349
8	6.604	VB	0.0399	2625.23071	1024.02734	0.05599
9	7.201	BB	0.0424	3.83950e4	1.42612e4	0.81881
10	7.725	BV	0.0420	4056.04556	1478.09326	0.08649
11	7.781	VB	0.0491	6311.43115	2096.62793	0.13460
12	8.196	BB	0.0491	2.81399e5	8.85710e4	6.00107
13	8.441	BP	0.0491	5642.98633	1822.01196	0.12034
14	8.831	PB	0.0531	5372.72852	1490.21008	0.11458
15	9.348	BP	0.0541	4.13679e5	1.14511e5	8.82207
16	9.997	BP	0.0503	9.53168e4	2.98522e4	2.03272
17	10.299	BV	0.0459	2.15391e4	6802.04395	0.45934
18	10.509	VB	0.0511	1.28429e4	3936.71069	0.27389
19	11.489	BB	0.0498	5767.81543	1826.76514	0.12300
20	11.704	BB	0.0503	2034.89209	619.49945	0.04340
21	13.334	VB	0.0536	1.70180e4	4889.50879	0.36302
22	35.731	PB	0.0589	2564.13745	661.40808	0.05469
23	16.289	BB	0.0636	7.33856e4	1.71709e4	1.56501
24	17.048	BP	0.0626	7.22502e4	1.76927e4	1.54080
25	17.509	BV	0.0882	2.40537e6	3.78367e5	51.29658
26	17.673	VB	0.0544	1.53750e4	4335.92383	0.32789
27	18.640	PP	0.0567	4315.57471	1127.23657	0.09203
28	19.131	PB	0.0953	6.79001e4	7404.51172	1.01934
29	20.251	BV	0.0666	2.73172e4	7301.37695	0.58256
30	20.341	VB	0.0564	1.30359e4	3426.96265	0.27800
31	20.526	BV	0.0619	3748.42212	933.17688	0.07991
32	20.660	VP	0.0523	1.62767e4	4835.62109	0.34712
33	21.044	BP	0.0496	2881.11206	804.14850	0.06144
34	21.483	BP	0.0570	9435.32520	2511.78687	0.20122
35	21.686	PP	0.0670	4289.74023	760.43774	0.09148
36	21.881	VP	0.0698	2.16543e4	5348.90291	0.46177
37	22.496	BV	0.0603	2682.54077	690.55219	0.05721
38	22.733	VV	0.0590	2.10024e5	4.62280e4	4.47894
39	22.935	VB	0.0620	1.39702e5	3.54340e4	2.97922
40	23.930	BB	0.0630	4617.43066	1098.02874	0.09847
41	24.285	BP	0.0614	4.28282e4	1.09960e4	0.91335
42	24.476	BB	0.0670	6815.48145	2485.28906	0.14540
43	24.884	PV	0.0689	2.30811e4	5909.27070	0.49222
44	25.013	VB	0.0626	1.49979e5	3.78097e4	3.19944
45	25.304	PB	0.0581	6137.13965	1987.29578	0.13096
46	25.610	BP	0.0626	1.09675e4	2880.99780	0.23399
47	26.683	PP	0.0712	3667.15894	774.43170	0.07821
48	26.952	PB	0.0669	6561.12793	1274.21338	0.11860
49	27.734	PB	0.0677	9651.86719	2377.33325	0.20583
50	28.755	BP	0.0688	3383.59717	720.25140	0.07216
51	28.992	BB	0.0604	1.70105e4	4375.78320	0.36276
52	30.461	BB	0.0642	6463.17334	1526.99902	0.13741
53	30.637	Bb	0.0557	2214.16968	634.68811	0.04722
54	31.341	BP	0.0561	1.78076e4	4817.12012	0.37976
55	32.145	PB	0.0673	5037.12500	1082.31750	0.10742
56	33 326	PB	0.0631	1.07605e4	2661.79053	0.22926
57	38.255	PB	0.0669	4971.22510	1140.32727	0.10602
58	40.209	BB	0.0942	7727.51904	1033.52136	0.16480

Totals : 4.68913e6 1.04977e6

Results obtained with enhanced integrator!

Linalool.

The unwanted peak.

Data File C:\HPCHEM\1\DATA\071204\S-30323.D
CORIANDER

Sample Name: CORIANDER

Injection Date	: 7/12/2004 12:38:17 PM	Seq. Line	: 15
Sample Name	: CORIANDER	Location	: Vial 15
Acq. Operator	: JOHN	Inj	: 1
		Inj Volume	: 3.1 µl
Sequence File	: C:\HPCHEM\1\SEQUENCE\071204.S		
Method	: C:\HPCHEM\1\METHODS\METHSTDA.M		
Last changed	: 6/25/2004 1:59:30 PM by JOHN		

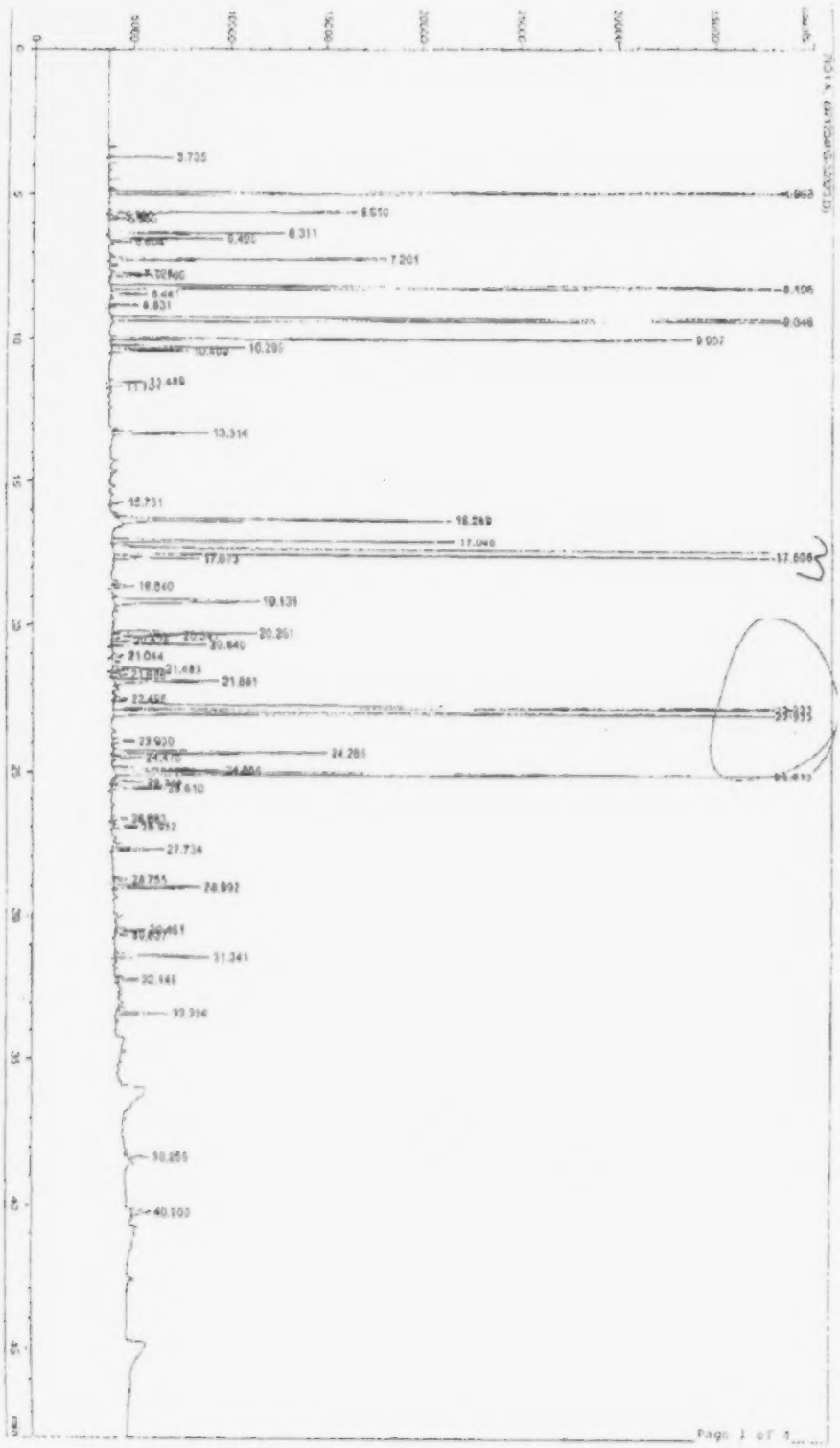
Signal 2: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	4.436	PP	0.0330	1561.40149	760.01105	0.03748
2	6.072	BB	0.0391	6330.46826	2616.99656	0.15677
3	6.477	BB	0.0372	4511.36133	1847.45972	0.10830
4	7.128	PP	0.0483	3346.38396	1076.52246	0.08033
5	7.350	pn	0.0449	2.60467e5	8.52517e4	5.77206
6	7.705	BB	0.0487	3.04359e4	9006.80273	0.73063
7	8.066	DB	0.0554	1969.80664	542.35034	0.04729
8	8.255	BP	0.0455	1.36151e4	4742.50398	0.32684
9	8.415	PB	0.0464	2.06942e4	7099.29932	0.49678
10	8.632	BB	0.0561	5.00941e4	1.29550e4	1.20254
11	9.102	BB	0.0586	2610.93726	668.11920	0.06268
12	9.468	PV	0.0403	3625.06519	1237.13220	0.08222
13	9.560	VB	0.0484	8.26640e4	2.65013e4	1.98440
14	9.861	BB	0.0519	2.47170e5	7.61093e4	5.93347
15	10.270	PB	0.0460	1889.58630	578.98029	0.04536
16	10.711	BP	0.0574	3.65304e5	9.59333e4	8.76933
17	11.296	PP	0.0508	2690.22803	831.09564	0.06458
18	11.598	PV	0.0578	2.29525e4	6112.13770	0.55099
19	11.373	VV	0.1161	2.09802e6	2.30648e5	50.36425
20	12.200	VP	0.0477	1992.70760	602.38062	0.04544
21	12.986	BB	0.0566	6.69193e4	1.83188e4	1.60644
22	13.216	BB	0.0704	5234.17725	1045.95605	0.12565
23	13.926	PB	0.0565	1.19481e4	3179.53003	0.28443
24	14.230	PP	0.0522	6056.00293	1851.91602	0.14539
25	14.558	BB	0.0576	9045.63965	2417.81860	0.21715
26	14.892	BB	0.0522	7.74499e4	2.30429e4	1.85923
27	15.685	BB	0.0671	1.03996e4	2163.86109	0.24965
28	15.933	BP	0.0632	1.77420e5	4.12431e4	4.25907
29	16.465	BV	0.0604	1.32420e5	3.32769e4	3.17881
30	16.533	VB	0.0446	2.33166e4	7863.20996	0.55975
31	16.701	BP	0.0518	3865.02783	1042.43115	0.08316
32	16.940	BV	0.0556	3.97453e4	1.08908e4	0.95411
33	17.074	VB	0.0544	6.36996e4	1.79505e4	1.52915
34	17.273	BB	0.0586	2.10609e4	5766.51172	0.50558
35	17.526	BB	0.0683	4881.90576	1068.02502	0.11719
36	17.845	BP	0.0501	3843.91162	1222.51355	0.09227
37	17.996	VB	0.0537	3.05140e4	8740.47559	0.73251
38	19.564	BP	0.0650	3316.56299	789.97803	0.07962
39	19.994	BV	0.1218	3.68860e4	4043.41943	0.88538
40	20.124	VP	0.0542	5.25010e4	1.49315e4	1.26031
41	20.937	BB	0.0674	3.68586e4	8055.33398	0.88481
42	21.847	BP	0.0590	6327.98243	1716.13928	0.15191
43	22.453	PV	0.0536	1.16410e4	3429.30249	0.27945
44	22.540	VB	0.0563	1.79155e4	5058.08936	0.43007
45	22.716	BV	0.0546	1.49805e4	4190.33496	0.35962
46	22.829	VP	0.0551	7613.49805	2107.56201	0.18277
47	23.571	PV	0.0569	8166.68994	2169.51221	0.19605
48	23.717	VB	0.0525	6132.68652	1812.04602	0.14722
49	25.129	PB	0.0715	6658.46533	1376.08362	0.15984
50	25.920	PB	0.0703	2931.95264	629.43567	0.07038
51	26.338	PP	0.0532	5564.88721	1616.03857	0.13359
52	27.735	PP	0.0585	1.11008e4	3123.38818	0.26648
53	27.886	VV	0.0566	9021.98145	2469.80981	0.21658
54	27.980	VP	0.0560	3628.80786	962.83459	0.00711
55	28.706	PB	0.0750	6662.21484	1386.81213	0.15993
56	29.963	PP	0.0676	7469.24561	1661.88330	0.17928
57	31.893	BP	0.0560	1.58909e4	4358.03711	0.38157
58	38.405	PB	0.0649	4321.52490	1030.66125	0.10374
Totals :				4.16570e6	8.11796e5	

Results obtained with enhanced integrator!

*** End of Report ***

Unexpected Peak.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7.201
8.105
9.007
10.299
13.314
16.269
17.606
19.131
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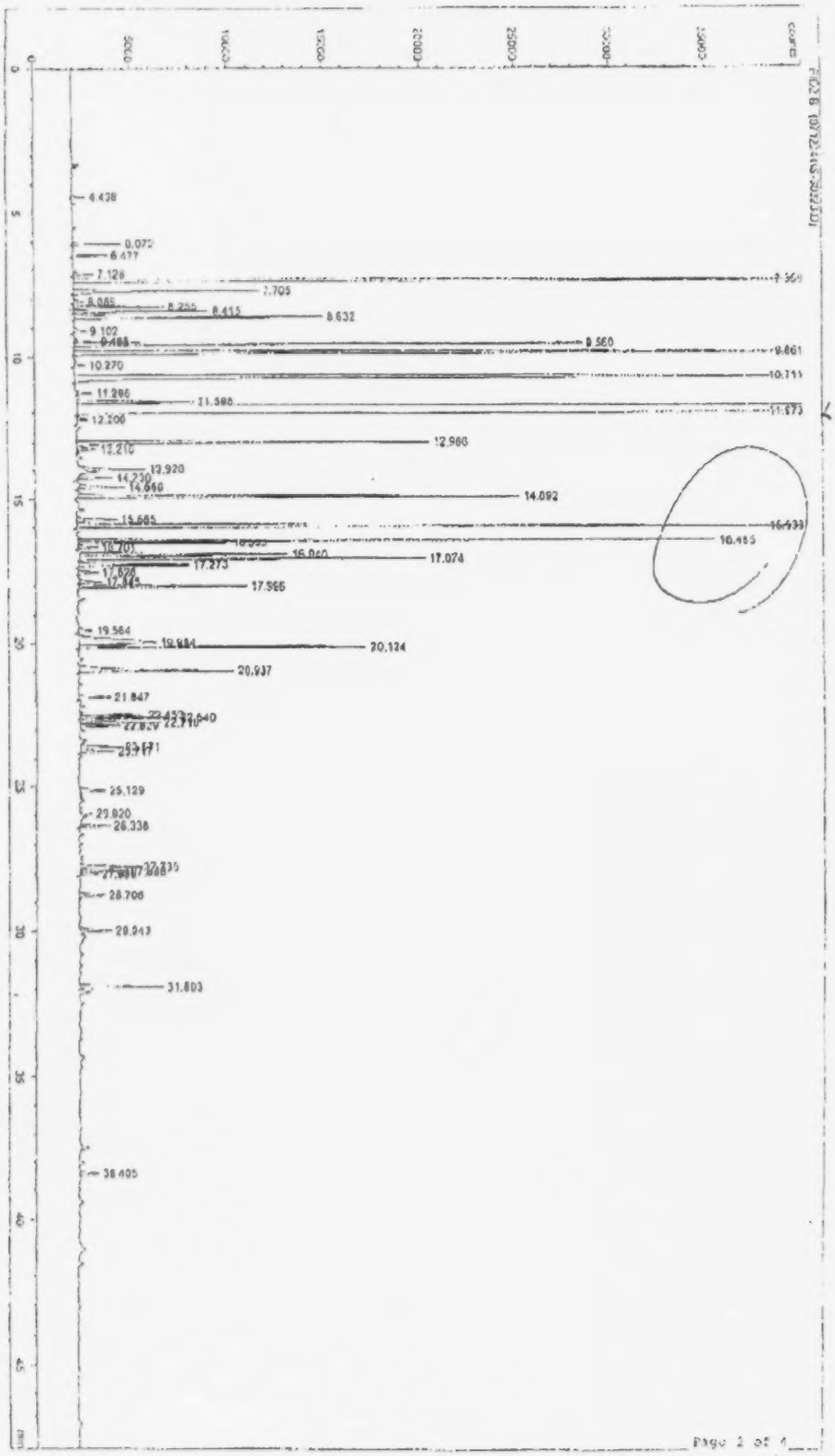
21.881
24.285
27.734
28.992
31.341
33.324
38.255
40.200
Page 1 of 1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7.705
8.632
9.102
9.560
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11.588
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13.210
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14.660
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17.074
17.273
17.966
19.564
20.124
20.937
21.847
25.129
25.920
26.338
28.706
29.943
31.803
38.405

Page 2 of 4

Q/C SAMPLE ANALYSIS REPORT

14-Jul-00

SR: S-30324

PRODUCT: ANISE OIL

SUPPLIER: SCHWEITZER ENTERPRISES

LOT#: LOT 16 & 17

PO#:

CONTRACT#:

PRIORITY: RUN EACH DRUM INDIVIDUALLY GIVE RESULTS TO GJ

DATE: 7/12/200

PURITY: (16) 85.36% (17) 89.57% Anethol

REFRACTIVE INDEX: 1.552 pass (1.549) low 1.553 min

SPECIFIC GRAVITY: 0.989 pass 0.977 low 0.978 min

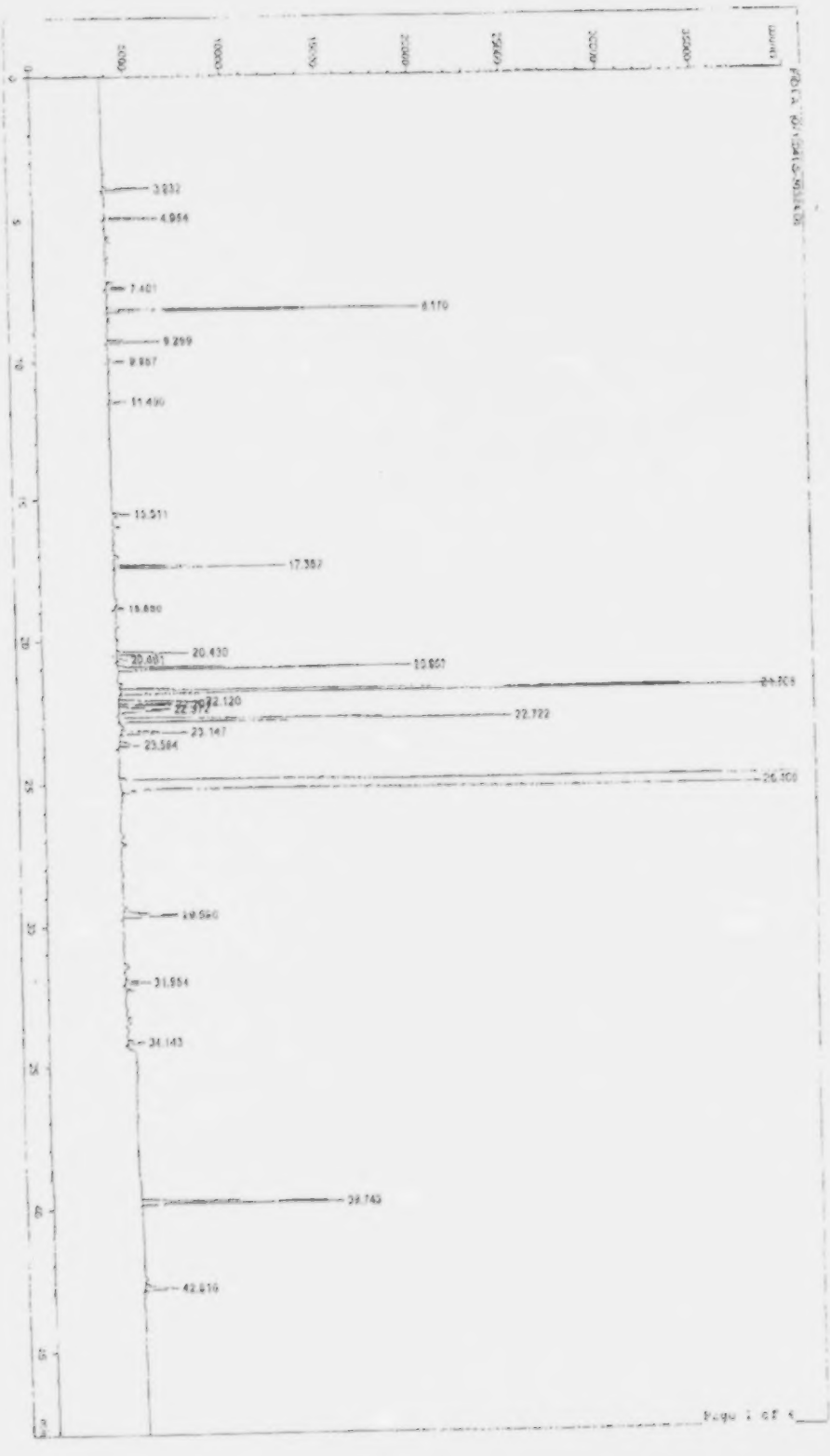
OPTICAL ROTATION: +1.4 pass +2.02 out 1.0 max

ODOR EVALUATION:

COLOR/COMMENTS: Pale yellow liquid

RECOMMENDATIONS:

FINAL APPROVAL: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8.170
9.299
9.957
11.490
13.511
17.387
18.600
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22.722
23.147
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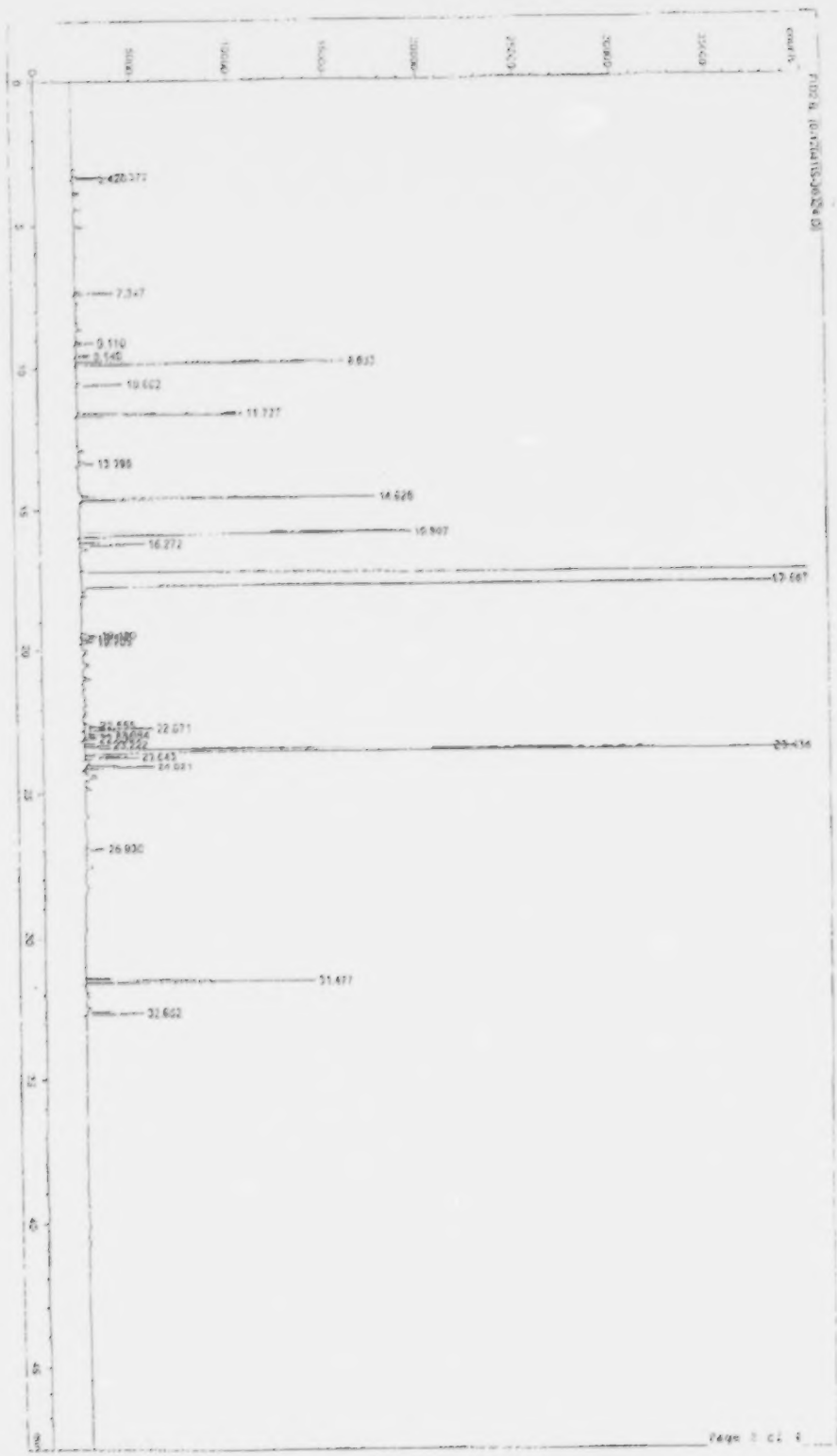
31.954
34.143
38.743
42.616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9.110
19.662
13.398
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16.272
22.071
26.930
31.477
32.602

Injection Date : 7/13/2004 12:30:50 AM
Sample Name : ANISE
Acq. Operator : JOHN

Seq. Line : 16
Location : Vial 16
Inj : 1
Inj Volume : 0.1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\0712041.S
Method : C:\HPCHEM\1\METHODS\METHSTDA.M
Last changed : 6/25/2004 1:59:30 PM by JOHN

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	3.932	BB	0.0392	6471.00586	2503.07764	0.10854
2	4.954	BB	0.0349	6309.80664	2837.42651	0.10584
3	7.401	BB	0.0436	3107.54150	1111.97192	0.05212
4	8.170	BB	0.0465	9.87169e4	1.64920e4	0.81716
5	9.289	BB	0.0492	8754.45508	2819.26514	0.14684
6	9.967	BB	0.0527	3482.55713	910.84839	0.05840
7	11.490	BB	0.0521	3295.53784	983.31403	0.05528
8	15.511	BB	0.0514	2929.55493	914.64886	0.04914
9	17.357	BB	0.0550	3.06751e4	8943.00879	0.51453
10	18.850	BB	0.0559	1886.08057	524.82300	0.03164
11	20.430	BB	0.0656	1.59940e4	3839.08159	0.26828
12	20.661	BB	0.0658	2489.37231	583.66162	0.04176
13	20.957	BB	0.0626	6.18163e4	1.54813e4	1.03688
14	21.769	BB	0.0719	1.68342e5	3.64630e4	2.82370
15	22.120	BV	0.0734	2.15284e4	4374.24316	0.36111
16	22.201	VV	0.0560	1.06819e4	2872.46509	0.17750
17	22.372	VB	0.0873	1.75359e4	2789.33447	0.29414
18	22.722	BB	0.0681	9.14317e4	2.08808e4	1.53364
19	23.147	BB	0.0648	1.53069e4	3517.40894	0.25675
20	23.584	BB	0.0778	6141.45117	1160.04163	0.10301
21	25.109	BB	0.1194	5.33992e6	5.79902e5	89.56975
22	29.590	BB	0.0925	1.81609e4	2922.27246	0.30462
23	31.954	BB	0.0729	6353.87256	1350.70959	0.10659
24	34.143	BB	0.0729	4292.83887	928.80060	0.07201
25	39.743	BB	0.0798	5.46024e4	1.06586e4	0.91588
26	42.816	BP	0.1012	1.10180e4	1809.17505	0.18923

Totals : 5.96175e6 7.27694e5

Results obtained with enhanced integrator!

Signal 2: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	3.372	VV	0.0256	3803.92870	2263.97607	0.07370
2	3.425	VB	0.0267	1981.74536	1174.39978	0.03839
3	7.347	BB	0.0444	5500.03174	1983.21252	0.10656
4	9.110	BP	0.0499	2898.94434	923.15594	0.05810
5	9.549	BB	0.0477	2235.47266	730.93896	0.04331
6	9.833	BB	0.0487	4.27267e4	1.39675e4	0.82767
7	10.662	BB	0.0479	7410.03662	2405.53174	0.14356
8	11.727	BB	0.0488	2.63581e4	8561.65137	0.51060
9	13.398	BP	0.0513	2642.65649	804.50800	0.05120
10	14.628	BP	0.0528	5.23519e4	1.53549e4	1.01426
11	15.907	BB	0.0802	9.08619e4	1.73346e4	1.76035
12	16.272	BB	0.0603	1.30954e4	3371.99390	0.25371
13	17.687	BB	0.1752	4.61445e6	3.20053e5	89.40015
14	19.480	BV	0.0530	2915.46094	850.56158	0.05648
15	19.596	VV	0.0588	2432.68457	633.60468	0.04713
16	19.703	VB	0.0563	2312.28099	638.39563	0.04480
17	22.555	BV	0.0525	2305.64014	680.16681	0.04467
18	22.671	VB	0.0593	1.37091e4	3608.20327	0.26560
19	22.894	BV	0.0562	5166.42090	1396.20667	0.10009
20	22.996	VB	0.0575	2368.39185	634.74719	0.04589
21	23.222	BV	0.0581	4985.51318	1318.59436	0.09665
22	23.430	VV	0.0666	1.65119e5	3.73908e4	3.19902
23	23.643	VB	0.0587	1.04332e4	2786.33789	0.20213
24	24.021	BB	0.0878	1.92345e4	3572.32422	0.37265
25	26.930	BB	0.0597	3903.18115	1018.50610	0.07562
26	31.477	BB	0.0631	4.82379e4	1.19976e4	0.93572
27	32.652	BB	0.0631	1.19727e4	2966.35840	0.23196

Data File C:\HPCHEM\1\DATA\071204\6-30324.D
ANISE 416

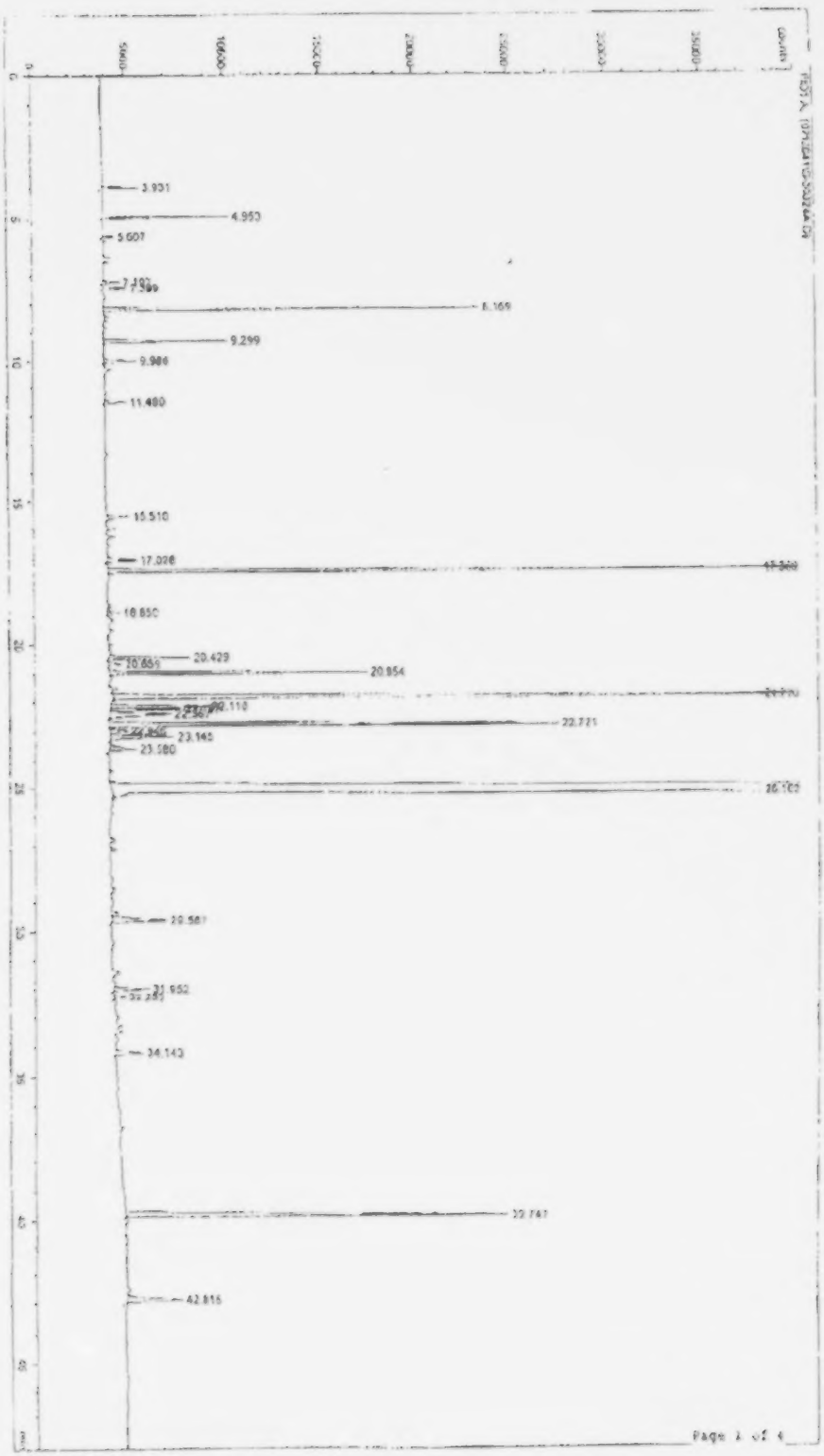
Sample Name: ANISE

Injection Date	: 7/13/2004 12:30:50 AM	Seq. Line	: 16
Sample Name	: ANISE	Location	: Vial 16
Acq. Operator	: JOHN	Inj	: 1
		Inj Volume	: 0.1 µl
Sequence File	: C:\HPCHEM\1\SEQUENCE\071204.S		
Method	: C:\HPCHEM\1\METHODS\METHOD1.M		
Last changed	: 6/25/2004 1:59:30 PM by JOHN		

Peak #	RetTime (min)	Type	Width (min)	Area counts*s	Height [counts]	Area %
Totals :				6.16186e6	4.5059146	

Results obtained with enhanced integrator!

*** End of Report ***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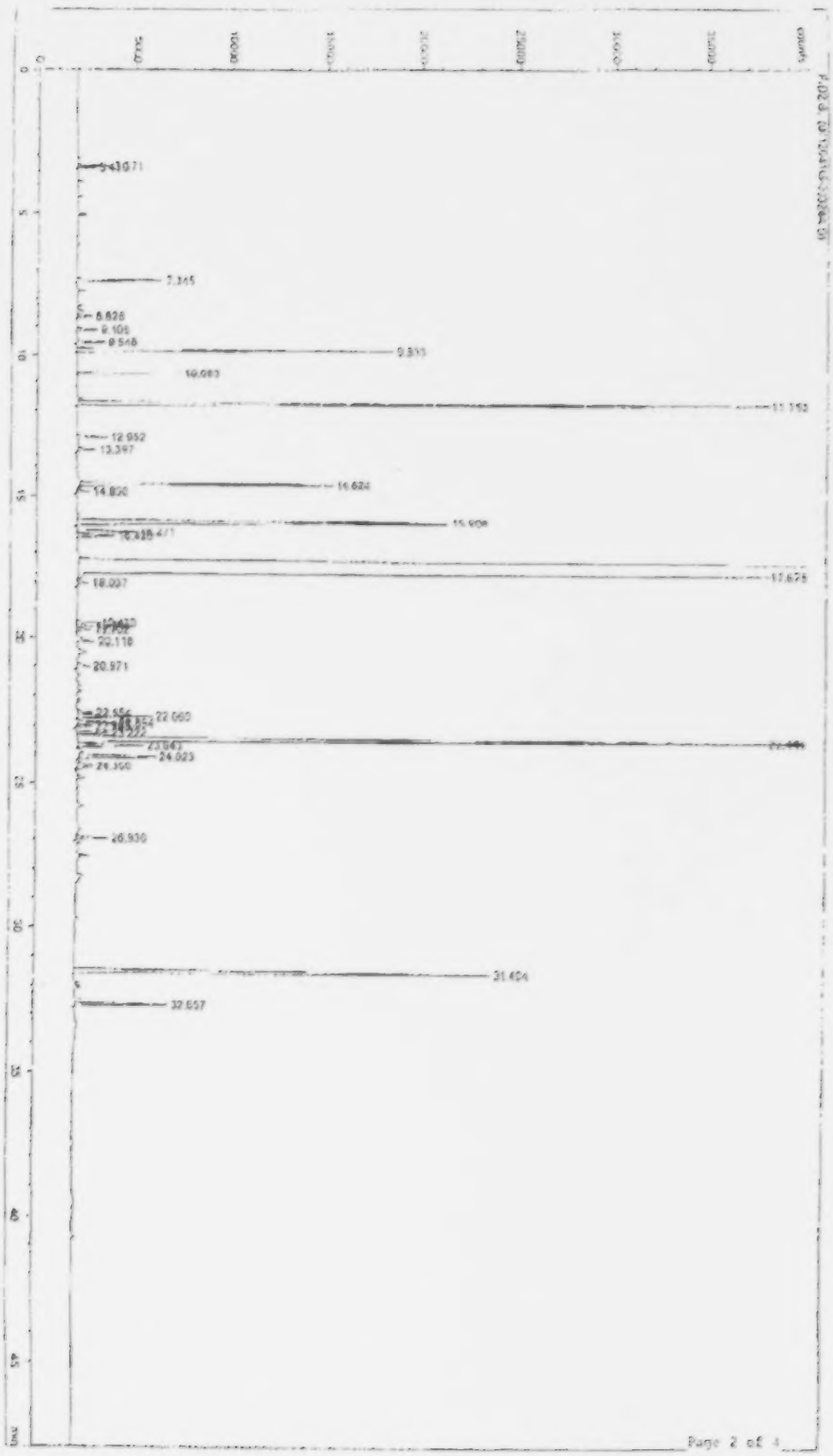
4.953
5.607
8.169
9.299
9.986
11.480
15.510
17.026
18.850
20.429
20.954
22.721
23.145
23.580
29.581
31.952
34.143
39.747
42.815


5430.71
7.345
8.626
9.105
9.548
9.833
10.093
11.152
12.952
13.397
14.800
16.624
15.908
17.625
18.097
20.118
20.971
22.660
24.023
26.930
31.404
32.657
Page 2 of 4

Data File C:\HPCHEM\1\DATA\0712041\8-30324A.D
ANISE 817

Sample Name: ANISE

Injection Date	: 7/13/2004 1:23:26 AM	Seq. Line	: 17
Sample Name	: ANISE	Location	: Vial 17
Acq. Operator	: JOHN	Inj	: 1
		Inj Volume	: 0.1 µl
Sequence File	: C:\HPCHEM\1\SEQUENCE\0712041.S		
Method	: C:\HPCHEM\1\METHODS\METKSTDA.M		
Last Changed	: 6/25/2004 1:59:30 PM by JOHN		

Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime (min)	Type	Width (min)	Area counts*s	Height (counts)	Area %
1	3.931	PB	0.0424	4996.44024	1914.41785	0.08345
2	4.953	BB	0.0351	1.43934e4	6440.22461	0.24038
3	5.607	BP	0.0399	1576.93672	619.02002	0.02634
4	7.197	BB	0.0425	2365.91431	867.98877	0.03918
5	7.399	BB	0.0438	3454.20093	1227.50562	0.05769
6	8.169	BB	0.0465	5.8103[illegible]e4	1.9636[illegible]e4	0.97030
7	9.299	BB	0.0482	1.95231e4	6289.67676	0.32606
8	9.986	BB	0.0515	5484.36230	1661.99475	0.09159
9	11.489	BB	0.0519	3755.16992	1127.24300	0.06272
10	15.510	PB	0.0513	3608.14355	1128.67041	0.06026
11	17.028	BP	0.0628	6260.84619	1529.67078	0.10456
12	17.369	BB	0.0550	1.44291e5	4.10583e4	2.40980
13	18.850	PP	0.0596	2356.51392	660.17780	0.03936
14	20.429	BB	0.0631	1.70887e4	4145.93799	0.28540
15	20.659	PB	0.0650	2952.95728	703.58807	0.04932
16	20.954	BB	0.0626	5.31704e4	1.33252e4	0.88800
17	21.770	PB	0.0684	1.84812e5	3.96689e4	3.08654
18	22.116	PV	0.0723	2.41135e4	4999.38721	0.40272
19	22.197	VV	0.0583	1.34301e4	3621.28320	0.22430
20	22.367	VP	0.0884	2.03630e4	3149.30591	0.34008
21	22.721	BB	0.0683	1.02660e5	2.33549e4	1.71453
22	22.946	BV	0.0889	5807.74561	916.69899	0.09700
23	23.145	VB	0.0702	1.51358e4	3259.20825	0.25278
24	23.580	PB	0.0757	7543.89209	1404.07190	0.12599
25	25.102	BB	0.1165	5.11108e6	5.65037e5	85.36011
26	29.507	BP	0.0944	1.81880e4	2013.34326	0.30376
27	31.952	BB	0.0726	8732.23438	1864.40613	0.14584
28	32.253	PP	0.0766	3030.12207	625.02954	0.05061
29	34.143	BB	0.0661	6807.90967	1497.12610	0.11370
30	35.747	BP	0.0806	1.94149e5	2.00416e4	1.73939
31	42.816	BP	0.0988	1.84537e4	2941.08252	0.30820
Totals :				5.98766e6	7.77426e5	

Results obtained with enhanced integrator!

Signal 2: FID2 B,

Peak #	RetTime (min)	Type	Width (min)	Area counts*s	Height (counts)	Area %
1	3.371	BV	0.0262	2775.46338	1682.67676	0.05353
2	3.419	VP	0.0266	1522.58026	908.29956	0.02936
3	7.345	BB	0.0431	1.21437e4	4407.77148	0.23419
4	8.628	BP	0.0469	2235.19849	746.40350	0.04311
5	9.108	BP	0.0500	3315.00708	1017.36523	0.06393
6	9.548	BB	0.0474	4201.16064	1385.48499	0.08102
7	9.833	BB	0.0477	5.04844e4	1.64878e4	0.97322
8	10.663	BB	0.0482	1.64524e4	5306.91309	0.31729
9	11.753	BB	0.0570	1.23774e5	3.60983e4	2.38700
10	12.952	MM	0.0535	5280.81396	1522.75586	0.10184
11	13.397	BP	0.0501	2835.88525	890.84979	0.05469
12	14.624	BB	0.0541	4.69756e4	1.30902e4	0.90597
13	14.866	PP	0.0511	2068.43286	632.77753	0.03989
14	15.908	BB	0.0786	9.80374e4	1.93160e4	1.90224
15	16.271	BV	0.0393	1.14813e4	3026.01758	0.22142
16	16.420	VB	0.0612	7321.81543	1850.08716	0.14120
17	17.675	BV	0.1713	4.40283e6	3.12608e5	84.90941
18	18.037	VB	0.0598	2148.45532	547.69379	0.04143
19	18.479	BV	0.0542	3582.66895	1040.34290	0.06909
20	18.596	VV	0.0595	3013.85718	773.07605	0.05812
21	19.702	VB	0.0607	2724.40161	681.15088	0.05254
22	20.118	BP	0.0470	2465.77344	821.87250	0.04755

Data File C:\HPCHEM\1\DATA\071204I\S-30324A.D
ANISE #17

Sample Name: ANISE

Injection Date : 7/13/2004 1:23:26 AM
Sample Name : ANISE
Acq. Operator : JOHN

Seq. Line : 17
Location : Vial 17
Inj : 1
Inj Volume : 0.1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\071204I.S
Method : C:\HPCHEM\1\METHODS\METHSTDA.M
Last change : 6/25/2004 1:59:30 PM by JOHN

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
23	20.971	BP	0.0637	2564.22388	626.90495	0.04945
24	22.554	PV	0.0518	2520.30013	756.91455	0.04861
25	22.669	VP	0.0601	1.48831e4	3946.47485	0.28702
26	22.894	PV	0.0577	7124.03955	1903.32019	0.13739
27	22.995	VP	0.0567	2695.09033	737.01050	0.05199
28	23.222	BV	0.0558	5884.45117	1567.83110	0.11348
29	23.441	VV	0.0689	1.84446e5	4.06507e4	3.55709
30	23.643	VB	0.0572	1.24759e4	3370.56561	0.24060
31	24.023	BP	0.0880	2.06964e4	3959.02197	0.39913
32	24.399	PB	0.1100	6872.18848	778.59949	0.13252
33	26.930	BP	0.0569	6200.11719	1610.65710	0.11957
34	31.494	BB	0.0646	9.38846e4	2.16631e4	1.81058
35	32.657	PP	0.0622	1.92286e4	4749.46533	0.37083
Totals :				5.18532e6	5.11062e5	

Results obtained with enhanced integrator!

*** End of Report ***

Q/C SAMPLE ANALYSIS REPORT

14-Jul-04

S#: S-30321

PRODUCT: CARAWAY OIL

SUPPLIER: SCHWEITZER ENTERPRISES

LOT#: LOTS 1 TO 8 & 14

PO#:

CONTRACT#:

PRIORITY: RUN EACH DRUM INDIVIDUALLY GIVE RESULTS TO GJ

DATE: 7/12/200

All lots on out high RI
high SG
Low OR
Light Caraway

	①	②	③	④	⑤	⑥	⑦	⑧	⑭	
PURITY:	71.97	70.25	69.40	69.29	70.31	69.86	69.94	69.12	71.22	
REFRACTIVE INDEX:	1.490	1.491	1.490	1.490	1.490	1.490	1.489	1.489	1.491	1.491
SPECIFIC GRAVITY:	0.930	0.928	0.927	0.927	0.928	0.928	0.926	0.921	0.929	0.91
OPTICAL ROTATION:	+65.8	+66.1	+67.6	+67.3	+66.7	+67	+67.6	+68	+66.4	70

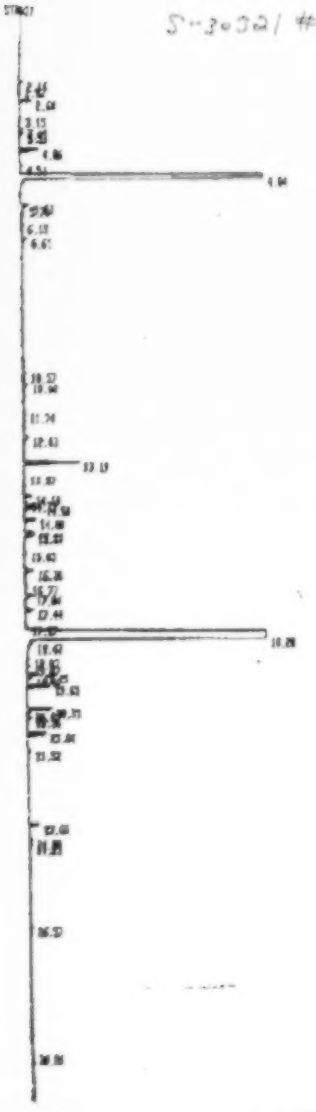
Caraway

ODOR EVALUATION: Ok

COLOR/COMMENTS: Pale yellow liquid

RECOMMENDATIONS:

FINAL APPROVAL:



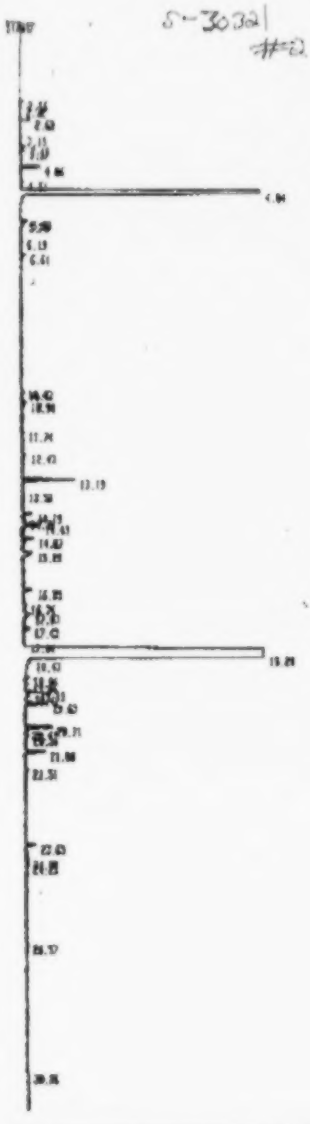
S-3032/ #1

RUN # 799
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 6

AREAS WT	AREA	TYPE	AM/WT	PMEAN
0.16	5871	BB	0.025	0.002
0.32	11594	0 PR	0.019	0.007
0.68	24751	0 PB	0.025	0.003
3.19	3385	PB	0.025	0.015
3.45	6253	PB	0.031	0.029
3.57	7939	BB	0.031	0.031
4.06	41868	PB	0.035	0.101
4.51	6863	BB	0.038	0.936
4.84	5242588	PB	0.971	23.434
5.63	16305	PV	0.035	0.035
5.79	7909	W	0.040	0.031
6.19	3119	PV	0.042	0.014
6.61	13898	PB	0.046	0.058
10.57	9697	W	0.049	0.037
10.99	10705	PB	0.047	0.046
11.74	4189	PB	0.050	0.018
12.43	16132	PB	0.061	0.067
13.19	134618	PB	0.044	0.598
13.52	4351	VB	0.052	0.019
14.19	25375	W	0.062	0.112
14.35	8719	PV	0.065	0.038
14.56	68794	VB	0.065	0.307
14.98	35530	PB	0.064	0.156
15.25	26283	W	0.046	0.115
15.34	27316	0 VB	0.052	0.120
15.83	4588	PB	0.075	0.022
16.36	26824	PB	0.064	0.118
16.77	9849	BV	0.071	0.044
17.04	32815	VB	0.080	0.145
17.44	24023	BB	0.065	0.106
17.67	4483	BP	0.063	0.019
18.01	1.4	PB	0.162	21.592
18.45	6585	BP	0.041	0.029
18.67	8899	BP	0.043	0.039
19.07	7822	PB	0.046	0.035
19.39	41483	PV	0.056	0.182
19.42	19291	VB	0.056	0.085
19.63	70435	PB	0.048	0.310
20.11	74903	PV	0.048	0.329
20.42	9825	W	0.055	0.043
20.56	10574	VB	0.053	0.046
21.01	56295	PB	0.049	0.244
21.32	9595	PB	0.052	0.041
22.05	32477	PV	0.052	0.143
24.08	8278	PV	0.058	0.036
24.83	8128	W	0.053	0.035
25.57	10586	PV	0.070	0.047
30.26	9112	BB	0.086	0.040

TOTAL AREA= 2.2731E+07
BAL FRACTION= 1

S-30321
#12

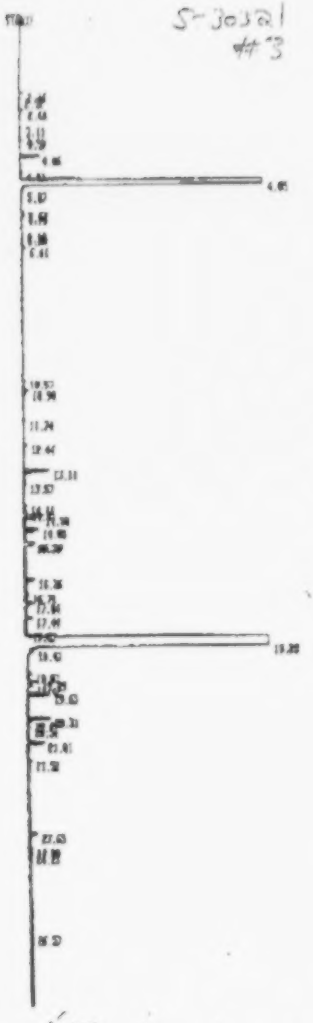


RUN # 717
MARKER ID: C1
SAMPLE # 14

RT	AREA	TYPE	HEIGHT	AREA%
2.16	6784	BB	0.838	0.809
2.62	2294	D PP	0.824	0.810
2.68	16484	D PB	0.825	0.978
3.15	3525	PB	0.833	0.815
3.45	8525	PB	0.831	0.837
3.52	7197	BB	0.831	0.891
4.86	43810	PB	0.835	0.188
4.51	6963	PB	0.851	0.838
4.84	5625388	PB	0.834	24.964
5.63	15337	PV	0.839	0.866
5.78	7963	VV	0.842	0.834
6.19	2851	PV	0.842	0.812
6.61	11645	PB	0.843	0.858
10.47	64477	BV	0.865	0.819
10.56	7962	VV	0.851	0.834
10.79	12871	BB	0.851	0.852
11.24	4659	BB	0.869	0.829
12.43	15688	PB	0.861	0.852
13.18	158879	PB	0.844	0.643
13.56	4337	VV	0.853	0.819
14.13	37488	PP	0.862	0.161
14.34	9107	PV	0.861	0.878
14.47	63451	VB	0.864	0.258
14.87	38579	PB	0.864	0.153
15.08	28819	VV	0.867	0.121
16.25	69869	VB	0.854	0.184
16.76	11717	PV	0.872	0.858
17.03	33881	VB	0.867	0.149
17.43	21796	BB	0.865	0.850
17.86	3791	BP	0.883	0.909
18.28	1.0344E+07	PB	0.162	29.752
18.43	9164	BP	0.842	0.833
18.86	8250	BB	0.845	0.835
19.06	6442	PB	0.843	0.859
19.29	37422	PV	0.856	0.161
19.42	13865	VB	0.855	0.867
19.63	69524	PB	0.847	0.258
20.31	10399	BV	0.848	0.854
20.61	7928	VV	0.855	0.832
20.56	11933	VB	0.854	0.851
21.00	60364	BB	0.849	0.853
21.51	9882	PB	0.852	0.870
22.63	324117	PB	0.852	0.135
24.06	9232	PV	0.856	0.869
24.23	8431	VV	0.863	0.836
24.57	7288	PP	0.863	0.852
28.26	7983	PB	0.888	0.838

TOTAL AREA= 2.XXXXE+07
MUL FACTOR= 1.0000E+00

S-30321
#3

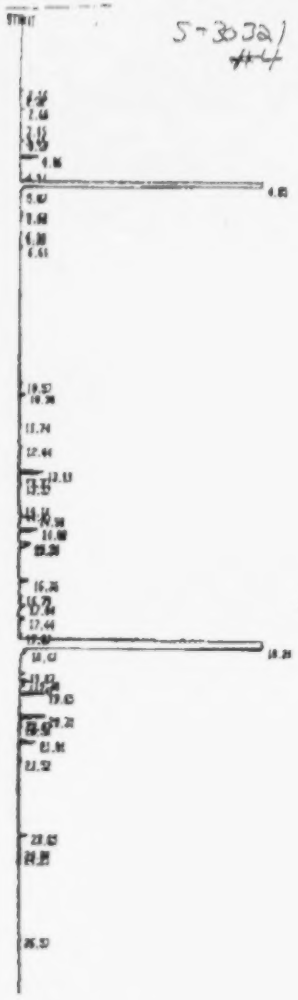


ESM # 212
[illegible] ID: C1
[illegible] NAME:
SAMPLE # 9

AREAS RT	AREA	TYPE	AR/HT	AREAS
2.16	15743	BB	0.965	0.824
2.32	17628	D VV	0.822	0.000
2.69	6397	B PB	0.823	0.000
3.15	3394	BB	0.974	0.014
3.45	2281	PB	0.928	0.010
3.57	7824	BV	0.931	0.030
4.06	42642	PB	0.935	0.176
4.51	4967	PB	0.847	0.021
4.85	6291888	PB	0.807	27.078
5.10	1779	PP	0.824	0.008
5.63	1075	PV	0.838	0.005
5.78	3581	VV	0.838	0.015
6.19	12282	PV	0.840	0.052
6.39	1824	VB	0.852	0.005
6.63	4887	BB	0.846	0.020
10.57	5716	VB	0.844	0.024
10.98	13372	PB	0.847	0.057
11.24	4826	BB	0.858	0.017
12.44	10381	PB	0.863	0.044
13.10	74577	PB	0.844	0.315
13.57	4364	VB	0.857	0.021
14.19	7333	PP	0.861	0.031
14.35	9476	PV	0.863	0.040
14.58	63981	VB	0.864	0.270
14.88	47648	BB	0.852	0.200
15.09	29282	VV	0.847	0.124
15.33	25615	D VB	0.858	0.108
16.25	27588	PB	0.854	0.117
16.78	8544	PV	0.859	0.036
17.04	33278	VB	0.886	0.140
17.44	25925	BB	0.867	0.110
17.97	7488	BV	0.872	0.031
18.28	1.6388E+07	PB	0.162	69.481
18.43	4481	BB	0.829	0.017
19.07	10540	BB	0.846	0.045
19.28	37854	PV	0.856	0.158
19.49	11824	VB	0.855	0.050
19.65	29113	PB	0.848	0.122
20.31	70641	BV	0.848	0.299
20.42	6933	VV	0.855	0.029
20.54	7369	VB	0.858	0.031
21.01	28401	PB	0.843	0.212
21.32	6987	PB	0.853	0.030
21.65	24748	PB	0.852	0.186
24.09	6816	PP	0.855	0.026
24.22	4413	PB	0.847	0.017
25.37	6589	PB	0.863	0.029

TOTAL AREA= 2.3635E+07
AREA FACTOR= 1.0000E+00

5-3021
#4

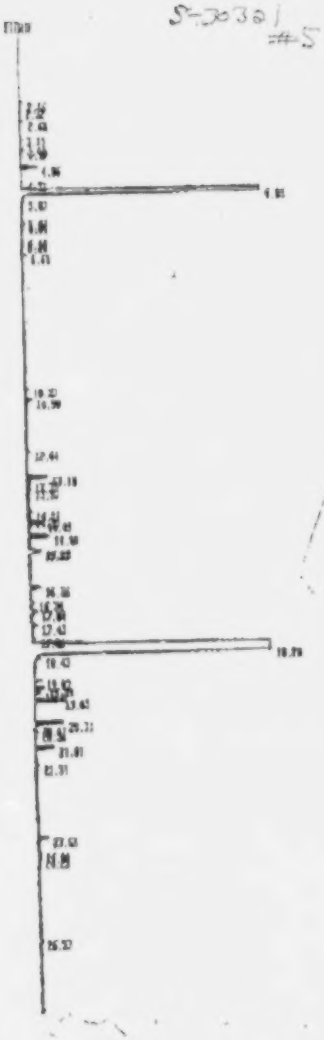


RUN # 218
WORKFILE ID= C1
WORKFILE NAME=
SAMPLE # 7

RT	AREA	TYPE	HEIGHT	AREA%
2.16	5358	PB	0.035	0.003
2.32	2189	O VP	0.024	0.009
2.64	6469	O BB	0.025	0.003
3.15	2642	BV	0.034	0.016
3.45	2258	PV	0.030	0.014
3.57	7269	BB	0.032	0.031
4.06	40891	PB	0.035	0.171
4.51	6936	PB	0.048	0.005
4.85	6264188	PB	0.077	25.856
5.07	1986	PV	0.035	0.008
5.63	5714	PV	0.048	0.024
5.79	9856	PV	0.048	0.025
6.19	3127	PV	0.048	0.013
6.28	7912	VV	0.062	0.033
6.61	10464	VB	0.042	0.043
10.57	8247	VB	0.049	0.035
10.98	19383	BB	0.050	0.079
11.74	3685	BB	0.053	0.015
12.44	2544	VB	0.052	0.043
13.19	67699	PB	0.044	0.287
13.37	4481	PV	0.048	0.017
13.57	6475	VB	0.056	0.027
14.19	6435	PV	0.061	0.028
14.25	8359	PV	0.053	0.035
14.58	64351	VB	0.064	0.273
14.98	61671	PB	0.053	0.254
15.29	35439	VV	0.046	0.143
15.34	31547	B VB	0.051	0.135
16.36	32184	PB	0.052	0.136
16.70	9635	PV	0.050	0.041
17.04	31166	VB	0.064	0.133
17.44	23282	BB	0.064	0.100
17.87	18226	BV	0.060	0.044
18.23	1.02	PB	0.161	69.256
18.43	6469	BV	0.026	0.026
19.07	14482	PB	0.046	0.062
19.38	36491	VV	0.056	0.156
19.62	16976	VB	0.054	0.073
19.83	75167	PB	0.048	0.321
20.31	79718	PV	0.048	0.341
20.42	7655	VV	0.035	0.033
20.56	2439	VB	0.039	0.032
21.01	53244	PB	0.049	0.228
21.52	8748	PB	0.049	0.037
23.03	28435	PB	0.032	0.122
24.00	6656	PV	0.057	0.028
24.23	6862	VV	0.060	0.029
25.57	3635	VV	0.032	0.015

TOTAL AREA= 2.34
MA FACTOR= 1.

S-30301
#5

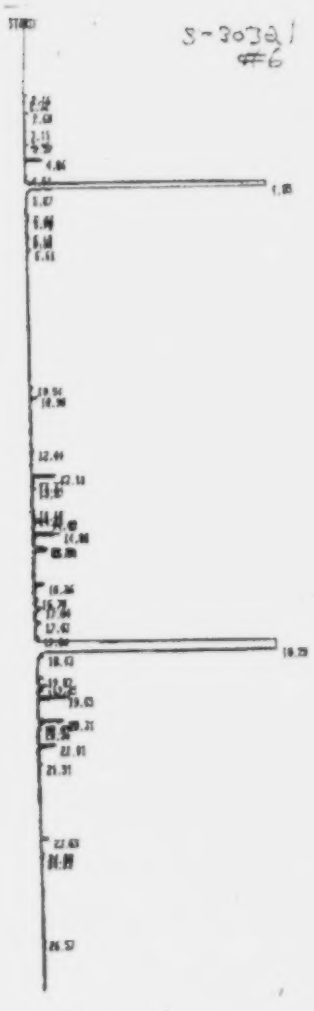


SIZE 9 215
WORKFILE ID= C1
WORKFILE NAME=
SAMPLE 6 12

AREA(N) HT	AREA	TYPE	AR/HT	AREA(D)
2.16	5365		0.000	0.000
2.32	1450	D	0.000	0.000
2.68	5363	D	0.000	0.000
2.35	2150		0.000	0.000
3.45	3257		0.000	0.000
3.52	6999		0.000	0.000
4.06	32208		0.000	0.000
4.51	6300		0.000	0.000
4.65	5835400		0.000	25.000
5.03	2481		0.000	0.000
5.63	4819		0.000	0.000
5.78	5453		0.000	0.000
5.75	7281		0.000	0.000
6.19	3147		0.000	0.000
6.28	4581		0.000	0.000
6.61	16193		0.000	0.000
10.57	8655		0.000	0.000
10.78	10914		0.000	0.000
12.44	3824		0.000	0.000
13.10	56473		0.000	0.000
13.32	4638		0.000	0.000
13.57	7228		0.000	0.000
14.18	11846		0.000	0.000
14.35	2846		0.000	0.000
14.49	57353		0.000	0.000
14.85	63179		0.000	0.000
15.28	33316		0.000	0.000
15.33	32954	D	0.000	0.000
16.36	33384		0.000	0.000
16.78	11169		0.000	0.000
17.04	28629		0.000	0.000
17.43	28459		0.000	0.000
17.86	14482		0.000	0.000
18.26	1.6882E+07		0.000	70.000
18.43	6348		0.000	0.000
19.07	21462		0.000	0.000
19.39	34485		0.000	0.000
19.62	18828		0.000	0.000
19.65	68500		0.000	0.000
20.31	10944		0.000	0.000
20.41	7800		0.000	0.000
20.56	2778		0.000	0.000
21.81	59749		0.000	0.000
21.91	9865		0.000	0.000
23.65	33581		0.000	0.000
24.08	7421		0.000	0.000
24.83	6825		0.000	0.000
25.32	6862		0.000	0.000

TOTAL AREA= 2.2760E+08
ALL FACTOR= 1.0000E+00

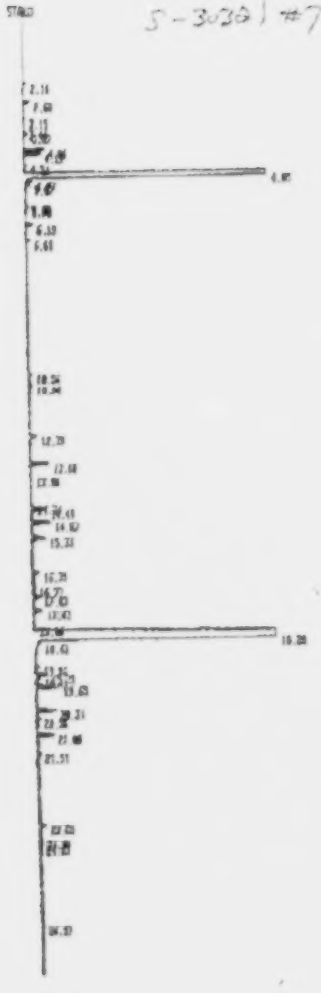
S-30321
#6



RUN # 711
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 0

RT	AREA	TYPE	AREA%	AREA%
2.16	4787	BB	0.026	0.024
2.32	1684	D PB	0.021	0.007
2.64	67288	D PB	0.025	0.025
3.15	3111	PB	0.024	0.014
3.45	21274	PB	0.029	0.087
3.57	2283	BB	0.031	0.021
4.06	38298	PB	0.035	0.165
4.51	4213	BB	0.055	0.017
4.65	6943486	PB	0.070	26.151
5.07	2113	PB	0.027	0.008
5.53	5885	PV	0.039	0.026
5.78	3529	VV	0.039	0.024
5.79	6411	VB	0.040	0.028
6.19	3315	BV	0.042	0.014
6.28	7283	VV	0.073	0.032
6.61	10446	PB	0.046	0.041
10.56	8569	VB	0.049	0.037
10.98	18546	BB	0.051	0.080
11.44	3065	PB	0.062	0.040
13.19	67280	PB	0.044	0.291
13.37	4114	PV	0.048	0.018
13.87	6573	VB	0.046	0.029
14.19	5235	PP	0.068	0.023
14.35	7613	PV	0.065	0.033
14.49	57618	VB	0.064	0.249
14.88	86793	PB	0.055	0.376
15.13	31134	PV	0.043	0.135
15.33	44918	VB	0.053	0.194
16.36	31467	PP	0.052	0.136
16.70	9405	PV	0.050	0.041
17.04	28286	VB	0.055	0.123
17.43	18998	BB	0.065	0.082
17.86	12283	BP	0.066	0.054
18.28	1.613354+07	PB	0.161	69.855
18.43	5893	BB	0.024	0.026
19.07	15919	PB	0.047	0.069
19.29	35397	PV	0.055	0.153
19.42	17805	VB	0.054	0.077
19.65	70529	PB	0.048	0.305
20.31	78696	PV	0.048	0.342
20.41	7355	VV	0.036	0.032
20.56	7712	VB	0.059	0.033
21.01	53362	PB	0.049	0.234
21.51	9516	PB	0.052	0.041
22.05	27529	PB	0.052	0.119
24.08	6981	PV	0.056	0.030
24.23	5464	VV	0.059	0.024
26.57	4425	PB	0.061	0.019

TOTAL AREA= 2.310E+07
MAL FACTOR= 1.000E+00



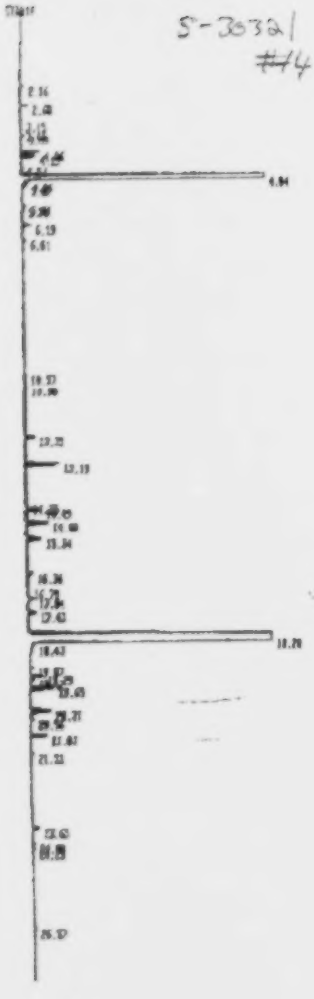
S-3038 #7

RUN # 214
NORMFILE 10: C1
NORMFILE NAME:
SAMPLE # 11

RT	AREA	TYPE	GRACT	AREAR
2.16	7171	PB	0.020	0.031
2.69	61330	B PB	0.005	0.043
3.15	3221	PB	0.006	0.014
3.49	2003	PB	0.010	0.009
3.57	8416	BB	0.001	0.036
4.06	47244	PB	0.006	0.204
4.19	38852	PB	0.002	0.168
4.36	4267	PB	0.011	0.019
4.85	6294888	PB	0.005	26.794
4.97	10138	BP	0.001	0.044
5.07	8828	PB	0.004	0.038
5.63	5491	PP	0.008	0.024
5.78	6284	VV	0.001	0.027
6.15	17267	PB	0.008	0.075
6.61	10467	PB	0.007	0.045
10.34	6885	PB	0.003	0.030
10.39	2438	PB	0.002	0.011
12.35	25127	PB	0.005	0.112
13.10	54282	PB	0.005	0.234
13.36	6882	PB	0.006	0.030
14.34	7298	PP	0.004	0.032
14.43	62438	PB	0.003	0.269
14.60	64188	PB	0.008	0.277
15.33	65556	PB	0.006	0.281
16.28	19579	BB	0.001	0.085
16.37	6838	PP	0.003	0.029
17.80	33535	VB	0.004	0.144
17.91	34743	BP	0.006	0.150
17.96	2581	PP	0.004	0.011
18.28	1.61985+07	PB	0.140	69.436
18.43	3420	BP	0.030	0.015
19.06	2015	BB	0.040	0.009
19.29	40539	PB	0.005	0.175
19.42	10886	VB	0.006	0.056
19.65	28298	PB	0.009	0.314
20.31	61339	PB	0.008	0.265
20.56	6794	VB	0.001	0.030
21.08	49681	PB	0.005	0.214
21.31	7574	PB	0.002	0.033
21.65	21851	PB	0.005	0.093
24.08	6495	PV	0.003	0.028
24.15	3221	VV	0.004	0.013
26.57	5593	BV	0.004	0.024

TOTAL AREA= 2.31[illegible]+07
NAL FACTOR= 1.[illegible]+00

S-3032
#14



RUN # 716
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 13

RETENTION TIME	AREA	TYPE	AMOUNT	AREA%
2.16	5126	PB	0.006	0.002
2.69	12827	D PB	0.005	0.003
3.15	2354	PB	0.007	0.001
3.45	8129	PB	0.028	0.005
3.57	7259	BB	0.001	0.002
4.06	414978	PB	0.035	0.102
4.19	33410	BB	0.026	0.147
4.51	3925	BB	0.049	0.017
4.84	5484499	PB	0.072	24.625
4.96	9524	BB	0.031	0.042
5.07	9539	PB	0.025	0.030
5.61	6973	PB	0.030	0.031
5.79	4771	VV	0.029	0.021
6.19	28857	PB	0.041	0.030
6.61	18694	PB	0.040	0.038
10.32	4249	PB	0.040	0.021
10.50	2457	BB	0.051	0.033
12.25	36833	PB	0.062	0.162
13.19	93128	PB	0.044	0.408
14.35	7897	PB	0.064	0.035
14.49	57998	VB	0.064	0.255
14.98	71305	PB	0.052	0.314
15.34	42348	PB	0.079	0.220
16.36	16962	PB	0.052	0.071
16.70	3698	BV	0.069	0.002
17.04	34487	VB	0.066	0.150
17.43	30383	BV	0.065	0.133
18.25	1.6233E+07	PB	0.160	71.225
18.67	8186	BB	0.029	0.005
19.07	7897	PB	0.045	0.033
19.29	43869	PV	0.056	0.193
19.42	12561	VB	0.052	0.055
19.65	71783	PB	0.049	0.315
20.31	81998	PV	0.063	0.359
20.56	19835	VB	0.058	0.090
21.01	49118	PB	0.041	0.216
21.51	7559	PB	0.032	0.033
23.65	81184	PB	0.035	0.033
24.98	7277	PV	0.052	0.032
24.23	9213	VV	0.069	0.040
26.57	6923	VV	0.071	0.030

TOTAL AREA= 2.2778E+07
MUL FACTOR= 1.0000E+00

Q/C SAMPLE ANALYSIS REPORT

14-Jul-04

S#: S-30322

PRODUCT: DILL OIL

SUPPLIER: SCHWEITZER ENTERPRISES

LOT#: LOT 9 TO 13

PO#:

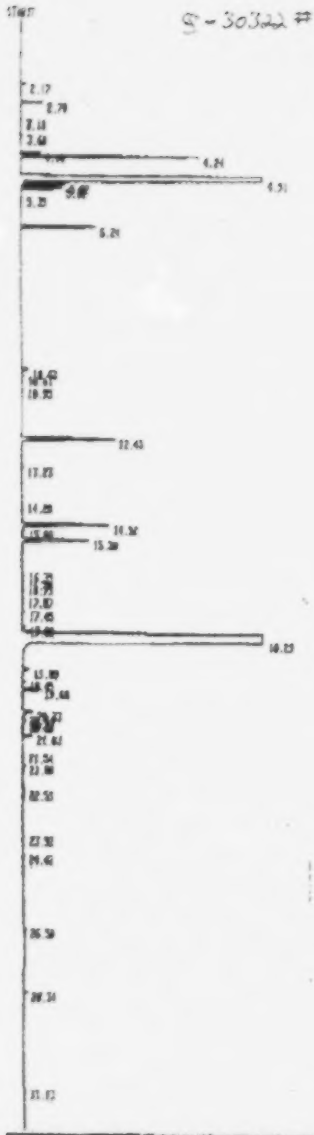
CONTRACT#:

PRIORITY: RUN EACH DRUM INDIVIDUALLY GIVE RESULTS TO GJ

DATE: 7/12/200

PURITY:	⑨ 59.40	⑯ 59.9	⑰ 63.54	⑫ 63.26	⑬ 65.98 light 60 max
REFRACTIVE INDEX:	1.488	1.488	1.489	1.489	1.490 dk.
SPECIFIC GRAVITY:	0.918	0.918	0.922	0.922	0.925 K out 0.915 ma
OPTICAL ROTATION:	+68.7	+68.4	+67	+66.5	+66.3 K 70 min
ODOR EVALUATION:					
COLOR/COMMENTS:	Pale yellow liquid				
RECOMMENDATIONS:					
FINAL APPROVAL:					

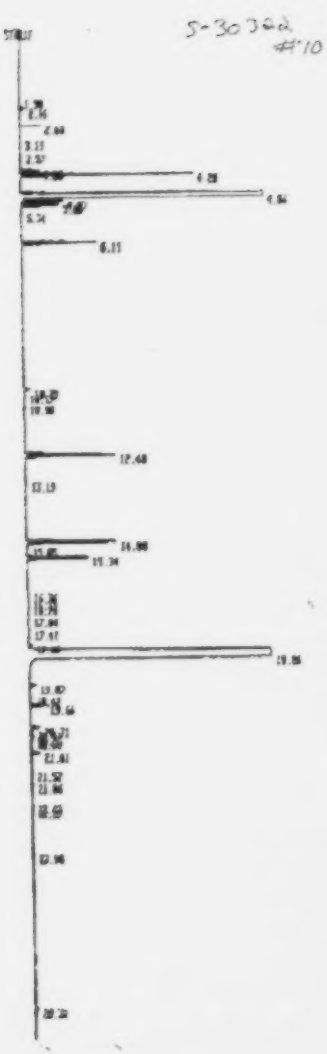
S-30322 #9



RUN # 704
WORKFILE 10: C1
MEASFILE NAME:
SAMPLE # 1

RT	AREA	TYPE	AR/MT	AR%/MT
2.17	8686	PV	0.027	0.035
2.79	32439 0	PB	0.025	0.159
3.18	1848	PV	0.030	0.008
3.68	4114	PB	0.031	0.018
4.18	47864	PP	0.036	0.205
4.24	424286	PB	0.037	1.829
4.91	7254588	PB	0.041	31.180
5.02	84775	BP	0.033	0.381
5.12	63863	PB	0.038	0.288
5.39	2463	BB	0.029	0.011
6.24	153960	PB	0.040	0.628
10.43	21784	BV	0.054	0.093
10.61	5088	VB	0.053	0.022
10.95	3569	BB	0.051	0.015
12.45	270619	BB	0.045	1.160
13.23	6367	BV	0.054	0.028
14.26	3382	BP	0.054	0.015
14.52	281938	PB	0.050	1.205
15.09	3582	BP	0.042	0.015
15.36	222218	PB	0.052	0.953
16.39	8999	BV	0.059	0.039
16.58	6979	VV	0.047	0.030
16.73	4119	VV	0.086	0.018
17.07	3424	BB	0.067	0.022
17.45	8568	BV	0.079	0.037
17.88	9483	PB	0.068	0.041
18.23	3.4884E+07	PB	0.150	68.297
19.09	10453	PB	0.047	0.029
19.48	6921	PP	0.054	0.023
19.68	10885	PB	0.056	0.250
20.33	44282	PV	0.073	0.136
20.44	7913	VV	0.055	0.034
20.57	18233	VV	0.055	0.044
20.65	6985	VP	0.051	0.026
21.03	28711	VB	0.048	0.123
21.54	4233	PB	0.052	0.018
21.89	5612	PB	0.049	0.024
22.39	3888	BP	0.056	0.016
23.02	4749	VB	0.038	0.020
24.42	3581	VB	0.035	0.015
25.20	34646	PB	0.148	0.033
28.34	9559	PB	0.051	0.040
31.13	3899	BB	0.042	0.017

TOTAL AREA= 2.2396E+07
RUL FACTOR= 1.0000E+00



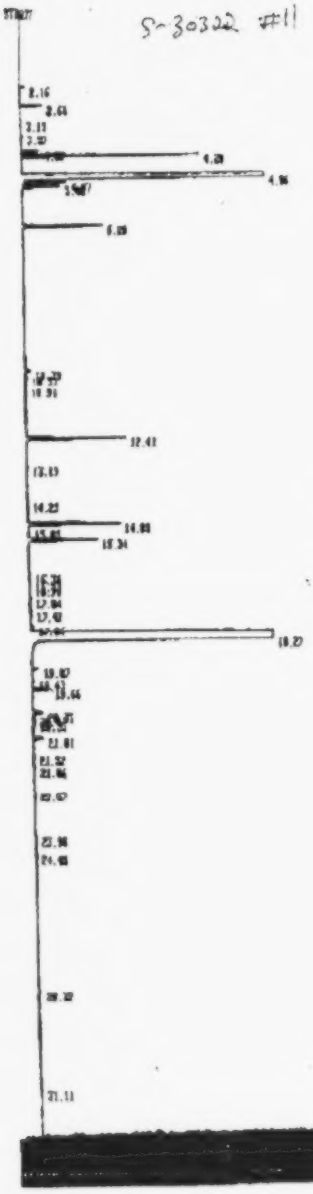
S-30392
#10

RUN 1 700
WORKFILE 10: C1
WORKFILE NAME:
SAMPLE 1 5

RT	AREA	TYPE	BR/MT	AREA%
1.98	1922	V PB	0.027	0.009
2.16	19139	BB	0.028	0.044
2.48	37040	V PB	0.025	0.161
2.15	1877	PV	0.030	0.009
2.52	2814	PB	0.031	0.012
4.06	47205	PP	0.035	0.206
4.28	405539	PB	0.037	1.749
4.95	7280580	VB	0.081	31.537
4.97	85849	BP	0.033	0.373
5.08	85243	PB	0.035	0.373
5.34	2000	BB	0.038	0.013
6.13	196198	PB	0.044	0.844
10.23	21435	BV	0.057	0.093
10.37	4728	VB	0.055	0.021
10.56	2500	PB	0.054	0.015
12.48	283230	PB	0.049	1.231
13.13	2241	BB	0.049	0.010
14.08	259898	PB	0.050	1.130
15.05	3478	BP	0.043	0.015
15.24	267576	PB	0.050	1.162
16.26	6349	BB	0.045	0.027
16.55	6253	PV	0.046	0.027
16.79	4417	VV	0.047	0.019
17.04	3638	BB	0.073	0.015
17.41	9524	BP	0.048	0.033
17.95	18521	BB	0.050	0.056
18.26	1.327782+07	BV	0.148	57.529
19.02	19398	PB	0.047	0.081
19.42	6427	PV	0.053	0.028
19.64	58167	PB	0.055	0.253
20.31	42048	BV	0.039	0.178
20.41	7944	VV	0.045	0.034
20.55	9991	VV	0.056	0.043
20.63	6246	VP	0.053	0.026
21.01	29363	PB	0.043	0.120
21.32	6165	PB	0.048	0.027
21.46	3363	PB	0.048	0.014
22.42	3254	PB	0.048	0.013
22.57	5435	VV	0.039	0.024
23.36	4255	VB	0.038	0.019
23.52	1622	PB	0.032	0.007

TOTAL AREA= 2.301E+07
WI, FACTOR= 1.000E+00

S-30322 #11

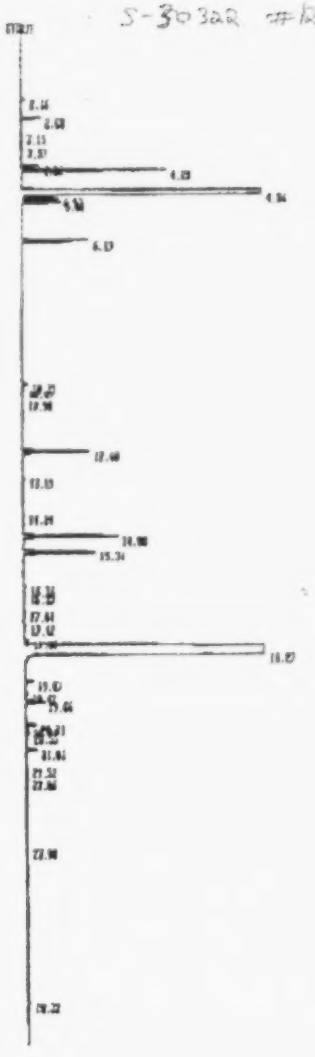


RUN # 785
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 2

RT	AREA	TYPE	AMT	AREA
2.16	8929	PV	0.926	0.870
2.63	30186	D PB	0.885	0.158
3.15	3164	PB	0.844	0.019
3.57	3809	BB	0.831	0.017
4.07	41439	PP	0.825	0.181
4.29	489598	PB	0.836	1.791
4.84	6548788	PB	0.877	27.734
4.97	82229	BP	0.843	0.301
5.88	88498	PP	0.925	0.352
6.09	139188	PB	0.948	0.529
10.39	19726	PV	0.956	0.086
10.57	5911	VB	0.953	0.026
10.91	3705	PB	0.851	0.016
12.41	302268	PB	0.848	1.328
13.19	6825	BV	0.859	0.030
14.15	3385	BP	0.854	0.015
14.88	255268	PB	0.858	1.270
15.85	3696	BP	0.843	0.016
17.34	226378	PB	0.852	0.992
16.36	6382	BV	0.961	0.028
16.555	7238	VV	0.879	0.032
16.79	3663	VV	0.856	0.016
17.04	5123	BP	0.872	0.022
17.42	6948	PP	0.877	0.030
17.86	11662	BP	0.868	0.051
18.37	1.4545E+07	PB	0.153	63.841
19.07	15974	PB	0.847	0.070
19.43	6565	PB	0.853	0.029
19.66	62463	PB	0.896	0.274
20.31	44486	PV	0.872	0.203
20.42	8636	VV	0.864	0.038
20.55	16970	VV	0.854	0.040
21.01	28144	PB	0.848	0.132
21.52	3961	PB	0.843	0.017
21.86	5821	PB	0.849	0.026
22.57	4491	BP	0.856	0.020
23.30	4364	VB	0.851	0.019
24.40	3642	BB	0.855	0.016
28.37	8746	PB	0.851	0.038
31.11	4411	PB	0.865	0.019

TOTAL AREA= 2.2891E+07
MUL FACTOR= 1.0000E+00

S-30322 #12



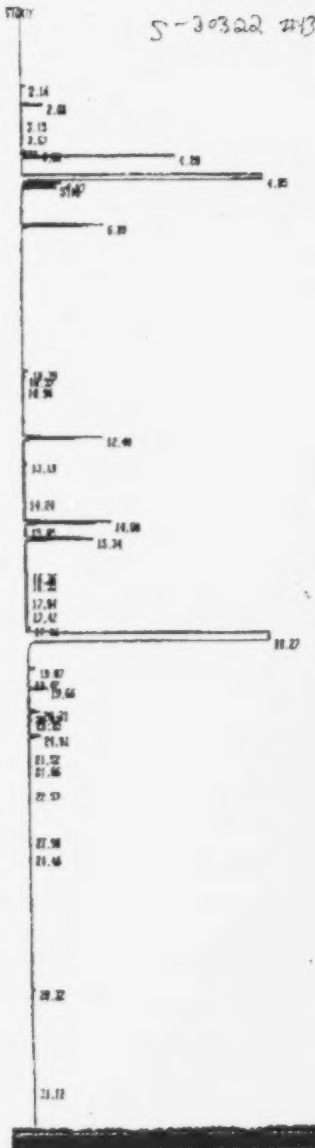
RUN # 2407
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 4

AREAS

RT	AREA	TYPE	WD/HT	AREA%
2.16	9464	PB	0.026	0.025
2.68	33676.0	PB	0.025	0.144
3.15	3813	VB	0.045	0.013
3.57	3962	PB	0.031	0.017
4.06	41830	PP	0.036	0.180
4.28	340340	PB	0.037	1.465
4.96	5611500	PB	0.070	22.460
4.97	74307	BP	0.023	0.323
5.08	20869	PB	0.025	0.221
6.19	175390	PB	0.048	0.755
10.20	19032	PV	0.057	0.080
10.57	4453	VB	0.053	0.020
10.78	40.37	BB	0.061	0.017
12.44	212290	PB	0.049	0.914
13.19	2672	BB	0.049	0.010
14.04	2184	PP	0.045	0.014
14.08	308160	PB	0.050	1.327
15.34	245190	PB	0.052	1.056
16.36	6490	BP	0.062	0.028
16.55	7305	PV	0.075	0.032
17.04	5240	BB	0.071	0.022
17.42	8311	BP	0.072	0.036
17.95	16431	PV	0.061	0.071
18.27	2.47906+07	VV	0.153	83.698
19.07	22732	PV	0.047	0.098
19.42	6587	PP	0.053	0.028
19.66	66283	PB	0.058	0.285
20.31	41367	PV	0.045	0.177
20.41	8365	VV	0.053	0.036
20.55	11899	VV	0.054	0.048
21.01	33231	PB	0.048	0.143
21.52	4171	PB	0.049	0.018
21.66	4638	PB	0.050	0.021
23.30	3748	VB	0.052	0.016
23.32	2486	PB	0.051	0.032

TOTAL AREA= 2.22216+07
AL FACTOR= 1.00000+00

5-30322 #3



RUN # 796
WORKFILE ID: C1
WORKFILE NAME:
SAMPLE # 3

AREA# RT	AREA	TYPE	AREA%	AREA#
2.16	7336	PB	0.026	0.162
2.60	37229	B PB	0.025	0.162
2.15	933	PB	0.029	0.004
3.57	3930	PB	0.031	0.017
4.06	38872	PV	0.035	0.165
4.28	367860	PB	0.036	1.588
4.85	3544000	PB	0.024	15.214
4.97	83285	BP	0.033	0.361
5.38	77542	PB	0.035	0.336
6.28	215188	PB	0.040	0.931
10.20	14834	VV	0.050	0.062
10.57	6463	VB	0.053	0.028
10.90	2301	PB	0.052	0.017
12.48	232348	PU	0.049	1.000
13.19	41944	BB	0.051	0.181
14.24	2629	PP	0.055	0.016
14.86	286588	PB	0.051	1.241
15.95	2171	BB	0.042	0.014
16.24	233229	PB	0.053	1.006
16.36	7271	BV	0.065	0.032
16.55	2063	VV	0.080	0.014
17.04	3824	BB	0.069	0.022
17.42	7738	BV	0.072	0.034
17.86	14118	PV	0.064	0.061
19.03	1.5243E+07	VV	0.155	65.790
19.07	20814	PB	0.047	0.090
19.47	6972	PP	0.055	0.030
19.66	69423	PB	0.056	0.300
20.31	43513	BV	0.063	0.188
20.42	8672	VV	0.054	0.037
20.55	11867	VV	0.065	0.051
21.01	35804	PB	0.048	0.152
21.32	4285	PB	0.047	0.018
21.86	3547	PB	0.049	0.024
22.37	3791	BP	0.058	0.016
23.98	4014	VB	0.052	0.017
24.48	3295	PB	0.054	0.014
28.32	5892	PB	0.051	0.025
31.12	4582	PB	0.069	0.020

TOTAL AREA= 2.3183E+07
RUN FACTOR= 1.0000E+00

APPENDIX III

Appendix III

Monographs of Essential Oil

Value, Ap-

ter Optical
nm tube.
Determina-
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Anise Oil

CAS: [8007-70-3]

DESCRIPTION

Anise Oil occurs as a colorless to pale yellow, strongly refractive liquid with the characteristic odor and taste of anise. It is obtained by steam distillation of the dried ripe fruit of *Pimpinella anisum* L. (Fam. Umbelliferae) or *Illicium verum* Hooker filius (Fam. Magnoliaceae).

> **Note:** If solid material has separated, carefully warm the sample until it is completely liquefied, and mix before using it.

Function Flavoring agent.

REQUIREMENTS

Identification The infrared absorption spectrum of the sample exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein.
Angular Rotation Between −2° and −1°.
Phenols Passes test.
Refractive Index Between 1.553 and 1.560 at 20°.
Solidification Point Not lower than 15°.
Solubility in Alcohol Passes test.
Specific Gravity Between 0.978 and 0.988.

TESTS

Angular Rotation Determine as directed under *Optical (Specific) Rotation*, Appendix IIB, using a 100-mm tube.
Phenols Prepare a 1:3 solution of recently distilled sample in 90% alcohol. It is neutral to moistened litmus paper, and adding 1 drop of ferric chloride TS to 5 mL of the solution produces no blue or brown color.
Refractive Index Determine as directed under *Refractive Index*, Appendix IIB, using an Abbé or other refractometer of equal or greater accuracy.
Solidification Point Determine as directed under *Solidification Point*, Appendix IIB.
Solubility in Alcohol Determine as directed under *Solubility in Alcohol*, Appendix VI, using 3 mL of 90% alcohol.
Specific Gravity Determine by any reliable method (see *General Provisions*).

Packaging and Storage Store in a cool place protected from light in full, tight containers that are made from steel or aluminum and that are suitably lined.

Annatto Extracts

INS: 160b CAS: [1393-63-1]

DESCRIPTION

Annatto Extracts occur as dark red solutions, emulsions, or suspensions in water or oil or as dark red powders. The extract is prepared from annatto seed, *Bixa orellana* L. (Fam. Bixaceae), using a food-grade extraction solvent. Bixin is the principal pigment of oil-soluble Annatto Extracts. Norbixin is the principal pigment of alkaline water-soluble Annatto Extracts. Commercial preparations are usually mixtures of bixin, norbixin, and other carotenoids.

Function Color.

REQUIREMENTS

Identification

A. Oil- and Water-Soluble Annatto Extracts Oil-soluble Annatto Extracts diluted with acetone exhibit absorbance maxima at 439–470, and 501 nm. Water-soluble Annatto Extracts diluted with water exhibit absorbance maxima at 451 to 455 nm and 480 to 484 nm.

B. Corr/Price Reaction (See *Chromatography*, Appendix IIA.) Prepare a small chromatography column by filling a 200- × 7-mm glass tube, stoppered with glass wool, with alumina (80- to 200-mesh) slurried in toluene so that the settled alumina fills about ⅔ of the tube. Using a rubber outlet tube and clamp, adjust the flow rate to about 30 drops/min.

Oil-Soluble Annatto Extracts Add to the top of the alumina column 3 mL of a solution containing sufficient sample.

Dillweed Oil, American Type

Dill Oil; Dill Herb Oil, American Type

CAS: [8006-75-5]

DESCRIPTION

Dillweed Oil, American Type, occurs as a light yellow to yellow liquid. It is the volatile oil obtained by steam distillation from the freshly cut stalks, leaves, and seeds of the plant *Anethum graveolens* L. (Fam. Umbelliferae). It is soluble in most fixed oils and in mineral oil. It is soluble, usually with opalescence or turbidity, in propylene glycol, but it is practically insoluble in glycerin.

Function Flavoring agent.

REQUIREMENTS

Identification The infrared absorption spectrum of the sample exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein.
Assay Not less than 28.0%, except in specific cases (see *Note*, below), and not more than 45.0%, by volume, of ketones as carvone.

> Note: Oil obtained from early season distillation may show a carvone content as low as 25.0% and a correspondingly lower specific gravity, lower refractive index, and higher angular rotation.

Angular Rotation Between +84° and +93° (see *Note*, above).
Refractive Index Between 1.480 and 1.485 at 20° (see *Note*, above).
Solubility in Alcohol Passes test.
Specific Gravity Between 0.884 and 0.900 (see *Note*, above).

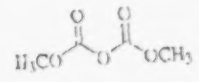
TESTS

Assay Determine as directed in the *Neutral Sulfite Method* under *Aldehydes and Ketones*, Appendix VI.
Angular Rotation Determine as directed under *Optical (Specific) Rotation*, Appendix IIB, using a 100-mm tube.
Refractive Index Determine as directed under *Refractive Index*, Appendix IIB, using an Abbé or other refractometer of equal or greater accuracy.
Solubility in Alcohol Determine as directed under *Solubility in Alcohol*, Appendix VI. One milliliter of sample dissolves in 1 mL of 90% alcohol, frequently with opalescence that may not disappear on dilution to as much as 10 mL.
Specific Gravity Determine by any reliable method (see *General Provisions*).

Packaging and Storage Store in a cool place protected from light in full, tight containers that are made from steel or aluminum and that are suitably lined.

Dimethyl Dicarbonate

Dicarbonic Acid, Dimethyl Ester; Dimethyl Pyrocarbonate; DMDC



$C_4H_6O_5$ Formula wt 134.09

INS: 242 CAS: [4525-33-1]

DESCRIPTION

Dimethyl Dicarbonate occurs as a clear, colorless liquid. Its solubility in water is 35 g/L at 20°, with decomposition. Its melting point is about 17°, and its flash point is 85°. It reacts quantitatively with water, producing carbon dioxide and methanol.

Caution: Dimethyl Dicarbonate is toxic if inhaled.

Function Preservative; antimicrobial.

REQUIREMENTS

Identification The infrared absorption spectrum of a neat dispersion of the sample between two sodium chloride plates exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein.
Assay Not less than 99.8% and not more than 101.5% of $C_4H_6O_5$.
Dimethyl Carbonate Not more than 0.2%.
Lead Not more than 1 mg/kg.

TESTS

Assay

1 N Di-n-butylamine Transfer 12.93 g of di-*n*-butylamine into a 100-mL volumetric flask, dilute to volume with toluene, and mix.

Procedure Transfer about 2 g of sample, accurately weighed, into a 250-mL beaker, and dissolve it in 100 mL of acetone. Add 25 mL of *1 N Di-n-butylamine* by pipet, allow the mixture to stand for 5 min, and titrate with *1 N* hydrochloric acid, determining the endpoint potentiometrically. Perform a blank titration (see *General Provisions*), and make any necessary correction. Calculate the percent Dimethyl Dicarbonate in the sample taken by the formula

$$100(B - A)0.134/W,$$

in which *B* and *A* are the volumes, in milliliters, of hydrochloric acid used for the blank and the sample, respectively; 0.134 is the milliequivalent weight of Dimethyl Dicarbonate; and *W* is the weight, in milligrams, of sample taken.

in which X_1 is the absorbance of the *Sample Solution* at 560 nm, and X_2 is the absorbance of the supernatant liquid at 560 nm.

2-Acetyl-4-(5)-tetrahydroxybutylimidazole (THI) [Class III (Ammonia Caramel) is the only class of Caramel color found to contain THI.] Because some countries have a THI limit of 25 ppm on an equivalent color basis, the following method for determining THI is provided.

2,4-Dinitrophenylhydrazine Hydrochloride (DNPH) Add 5 g of reagent-grade 2,4-dinitrophenylhydrazine to 10 mL of hydrochloric acid contained in a 100-mL Erlenmeyer flask, and gently shake the latter until the free base (red) is converted to the hydrochloride (yellow). Add 100 mL of ethanol, and heat the mixture on a steam bath until all of the solids have dissolved. Cool to room temperature, and after the solution has crystallized, filter off the hydrochloride. Wash the hydrochloride with ether, dry at room temperature, and store in a desiccator. Upon storage, the hydrochloride slowly converts to the free base. The latter can be removed by washing with purified (peroxide-free) dimethoxyethane.

DNPH Hydrochloride Reagent Prepare the reagent by mixing 0.5 g of *DNPH Hydrochloride* with 15 mL of 5% methanol in dimethoxyethane for 30 min. Store in a refrigerator at 4°. When properly prepared and stored, this reagent is stable for at least 3 months.

THI-DNPH Standard Add 0.5 g of *DNPH Hydrochloride* to 1 mL of hydrochloric acid, followed by 10 mL of ethyl alcohol, and heat on a steam bath until solution. Add 100 mg of THI to the hot solution—crystallization begins in a few minutes. Filter off the THI-DNPH when the suspension reaches room temperature. Obtain the *THI-DNPH Standard* by recrystallizing the THI-DNPH with a hydrochloric acid–ethyl alcohol mixture (1 drop of hydrochloric acid per 5 mL of ethyl alcohol). The yield is 70% to 80% based on the THI used. When stored in the refrigerator, the *THI-DNPH Standard* is stable for at least 1 year.

Stock THI-DNPH Solution Dissolve about 10 mg of *THI-DNPH Standard*, accurately weighed, in a 100-mL volumetric flask, and dilute to volume with absolute carbonyl-free methanol. Dilute a portion of this solution tenfold with methanol. The THI concentration, in milligrams per liter, of the *Stock THI-DNPH Solution* is 0.47 times that of the THI-DNPH. When stored in the refrigerator, the *Stock THI-DNPH Solution* is stable for at least 20 weeks.

Cation-Exchange Resin (Strong) Dowex 50 AG × 8, proton form, 100- to 200-mesh.

Cation-Exchange Resin (Weak) Amberlite CG AG 50 I, proton form, 100- to 200-mesh. (Sediment two or three times before use.)

Carbonyl-Free Methanol Add 5 g of Girard's Reagent P (Aldrich, or equivalent) and 0.2 mL of hydrochloric acid to 500 mL of methanol, and reflux for 2 h. Distill the refluxed methanol through a short Vigreux column, and store in tightly closed bottles.

Purified Dimethoxyethane Use distillation to purify dimethoxyethane from 2,4-dinitrophenylhydrazine in the presence of acid, and redistill it from sodium hydroxide. Immediately before use, pass it through a column of neutral alumina to remove peroxides.

Combination Column Connect a dropping funnel and two small columns, one fitted above the other, with 14/5-mm standard ground-glass joints. Use a 100-mL dropping funnel equipped with a Teflon stopcock, as the solvent reservoir. Fill the upper column [150 × 12.5 mm (id), filling height max 9 cm, bed height 50 to 60 mm; or 250 × 10 mm (id), filling height max 14 cm, bed height 80 to 90 mm], equipped with a 1-mm (id) capillary outlet, with *Cation-Exchange Resin (Weak)*. Fill the lower column [175 mm × 10 mm (id), bed height 60 mm], equipped with a 1-mm (id) capillary outlet and a Teflon stopcock, with *Cation-Exchange Resin (Strong)*.

Sample Preparation Dissolve 200 to 250 mg of sample, accurately weighed, in 3 mL of water. Quantitatively transfer the solution to the upper part of the combination column. Elute with water until a total of about 100 mL of water has passed through the column. Disconnect the upper column, and elute the lower column with 0.5 *N* hydrochloric acid. Discard the first 10.0 mL of eluate, and subsequently collect a volume of 35 mL. Concentrate the solution to dryness at 40° (15 mm Hg), then dissolve the syrup residue in 250 µL of carbonyl-free methanol, and add 250 µL of *DNPH Hydrochloride Reagent*. Transfer the reaction mixture (sample) to a septum-capped vial, and store for 5 h at room temperature.

Procedure Prepare a series of *THI-DNPH Standard Solutions* serially diluted from the *Stock THI-DNPH Solution*. Pipet 1, 2, and 5 mL, respectively, of the *Stock THI-DNPH Solution*, into separate 10-mL volumetric flasks, and dilute to volume with absolute, carbonyl-free methanol. Prepare a standard curve by injecting 5 µL of the *Stock THI-DNPH Solution*, and the serially diluted *THI-DNPH Standard Solutions* into a 250-mm × 4-mm (id), 10-µm LiChrosorb RP-8 HPLC column (Alltech Associates, Inc., or equivalent) fitted with an ultraviolet detector set at 385 nm. The mobile phase is 50:50 (v/v) methanol:0.1 *M* phosphoric acid. Inject 5 µL of sample into the column. Adjustments in the mobile phase composition may be needed as column characteristics vary among manufacturers. At a mobile phase flow rate of 2 mL/min and column dimensions of 250 × 4.6 mm, elute THI-DNPH at about 6.3 ± 0.1 min. Measure the peak areas. Calculate the amount of THI in the sample from the standard curve. (For THI limits greater than 25 mg/kg, prepare a series of *Standard THI-DNPH Solutions* in a range encompassing the expected THI concentration in the sample.)

Caraway Oil

CAS: [8000-42-8]

DESCRIPTION

Caraway Oil occurs as a colorless to pale yellow liquid with the characteristic odor and taste of caraway. It is a volatile

oil distilled from the dried, ripe fruit of *Carum carvi* L. (Fam: Umbelliferae).

Function Flavoring agent.

REQUIREMENTS

Identification The infrared absorption spectrum of the sample exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein.
Assay Not less than 50.0%, by volume, of ketones as carvone.
Angular Rotation Between +70° and +80°.
Refractive Index Between 1.484 and 1.488 at 20°.
Solubility in Alcohol Passes test.
Specific Gravity Between 0.930 and 0.910.

TESTS

Assay Determine as directed in the *Neutral Sulfite Method* under *Aldehydes and Ketones*, Appendix VI.
Angular Rotation Determine as directed under *Optical (Specific) Rotation*, Appendix IIB, using a 100-mm tube.
Refractive Index Determine as directed under *Refractive Index*, Appendix IIE, using an Abbé or other refractometer of equal or greater accuracy.
Solubility in Alcohol Determine as directed under *Solubility in Alcohol*, Appendix VI. One milliliter of sample dissolves in 8 mL of 80% alcohol.
Specific Gravity Determine by any reliable method (see *General Provisions*).

Packaging and Storage Store in full, tight containers in a cool place protected from light.

Carbon, Activated

DESCRIPTION

Carbon, Activated, occurs as a black substance, varying in particle size from coarse granules to a fine powder. It is a solid, porous, carbonaceous material prepared by carbonizing and activating organic substances. The raw materials, which include sawdust, peat, lignite, coal, cellulose residues, coconut shells, and petroleum coke, may be carbonized and activated at a high temperature with or without the addition of inorganic salts in a stream of activating gases such as steam or carbon dioxide. Alternatively, carbonaceous matter may be treated with a chemical activating agent such as phosphoric acid or zinc chloride, and the mixture carbonized at an elevated temperature, followed by removal of the chemical activating agent by water washing. Activated Carbon is insoluble in water and in organic solvents.

Function Decolorizing agent; taste- and odor-removing agent; purification agent in food processing.

REQUIREMENTS

Identification

A. Place about 3 g of powdered sample in a glass-stoppered Erlenmeyer flask containing 10 mL of dilute hydrochloric acid (5%), boil for 30 s, and cool to room temperature. Add 100 mL of iodine TS, stopper, and shake vigorously for 20 s. Filter through Whatman No. 2 filter paper, or equivalent, discarding the first portion of filtrate. Compare 50 mL of the subsequent filtrate with a reference solution prepared by diluting 10 mL of iodine TS to 50 mL with water, but not treated with carbon. The color of the carbon-treated iodine solution is no darker than that of the reference solution, indicating the adsorptivity of the sample.

B. Ignite a portion of the sample in air. Carbon monoxide and carbon dioxide are produced, and an ash remains.

Cyanogen Compounds Passes test.
Higher Aromatic Hydrocarbons Passes test.
Iodine Number Not less than 900.
Lead Not more than 10 mg/kg.
Water Extractables Not more than 4.0%.
The following additional *Requirements* should conform to the representations of the vendor: *Loss on Drying* and *Residue on Ignition*.

TESTS

Cyanogen Compounds Mix 5 g of sample with 50 mL of water and 2 g of tartaric acid, and distill the mixture, collecting 25 mL of distillate below the surface of a mixture of 2 mL of 1 *N* sodium hydroxide and 10 mL of water contained in a small flask placed in an ice bath. Dilute the distillate to 50 mL with water, and mix. Add 12 drops of ferrous sulfate TS to 25 mL of the diluted distillate, heat almost to boiling, cool, and add 1 mL of hydrochloric acid. No blue color is produced.
Higher Aromatic Hydrocarbons Extract 1 g of sample with 12 mL of cyclohexane in a continuous-extraction apparatus for 2 h. Place the extract in a Nessler tube and a solution of 100 µg of quinine sulfate in 1000 mL of 0.1 *N* sulfuric acid in a matching Nessler tube. The extract shows no more color or fluorescence than does the solution when observed under ultraviolet light.
Iodine Number[1]

Hydrochloric Acid Solution (5% by weight) Add 70 mL of concentrated hydrochloric acid to 550 mL of water, and mix well.

Potassium Iodate Solution (0.1000 *N*) Dry 4 or more grams of primary standard-grade potassium iodate (KIO_3) at 110° ± 5° for 2 h, and cool to room temperature in a desiccator.

[1]Portions of this text are adapted from "ASTM D 6607-94(1999)—Standard Test Method for Determination of Iodine Number of Activated Carbon." The original ASTM method is available in its entirety from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428; phone: 610-832-9585; fax: 610-832-9555; email: service@astm.org; website: <www.astm.org>.

APPENDIX IV

Appendix IV

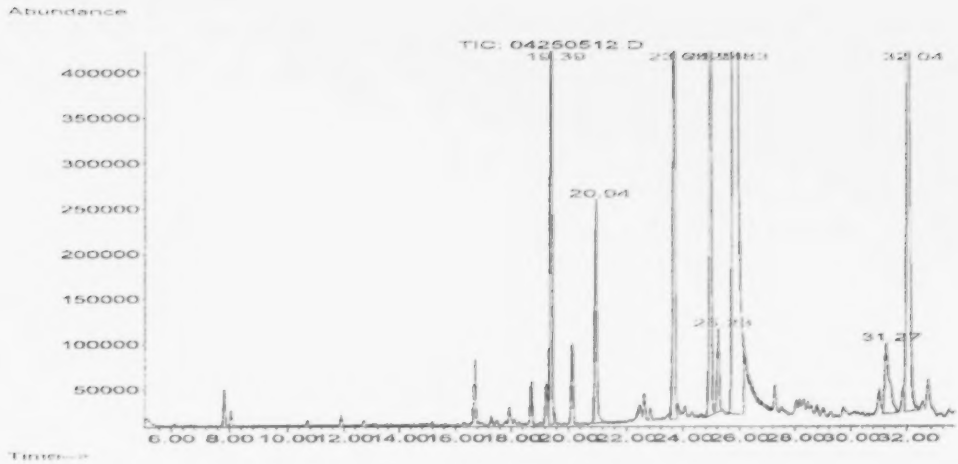
POS Final Testing

TEST AFTER FILTERING AND CHILLING:

Sample	Refractive Index	Specific Gravity	POS Initial Tests R.I.	POS Initial Tests S.G.	Buyers Initial Tests
dill #1	1.4889	0.9216			S.G. low 0.918 vs. 9.19
dill #2	1.4889	0.9201	not done		Optical Rotation
dill #3	1.4891	0.9206			68.7 vs. 70 min
caraway #1	1.4905	0.9261			R.I. 1.49
caraway #2	1.4905	0.9281	1.49	.928	(high – 1.48 ideal)
caraway #3	1.4905	0.9251			S.G. .928 vs. 0.91 ideal
coriander #1	1.4624	0.8668			
coriander #2	1.4621	0.8663			
coriander #3	1.4621	0.8693	1.4619	0.8702	All good readings
coriander #4	1.4621	0.8673			
coriander #5	1.4620	0.8678			
anise/coriander #1	1.5497	0.9769	1.55	.989	R.I. 1. 549 vs. 1.553
anise initial	1.5515	0.9804	1.553	.985	S.G. .997 vs. .978
anise #1	1.5541	0.9755			

1) Note that dill S.G. is now within the 0.919 minimum standard.
2) Caraway samples did not improve.
3) Coriander samples were all OK originally and no change for S.G. and R.I.
4) Anise process #1 (heat and stir) provided a sample that meets R.I. requirements and Specific Gravity.

ANISE INITIAL

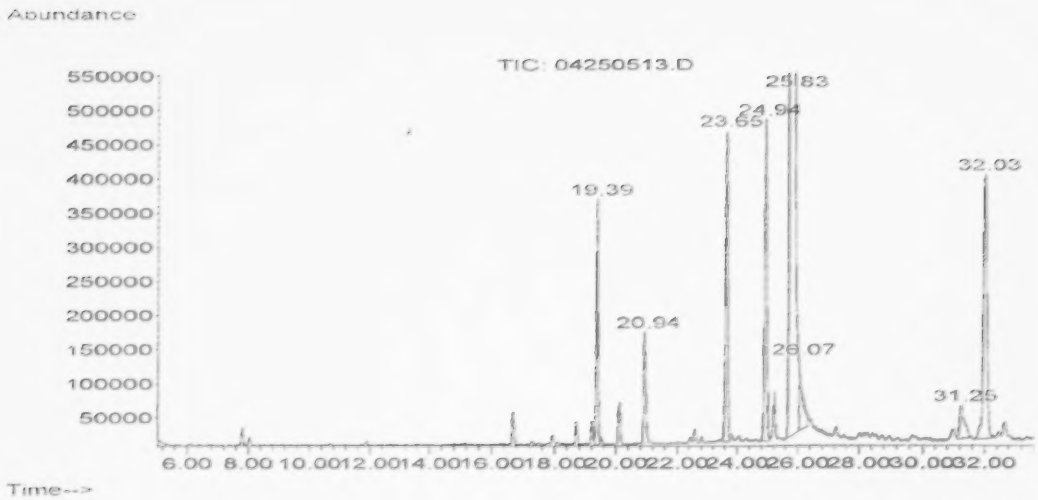


Anise initial

TIC: 04250512.D

Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	19.395	21480708	1.91	limonene	94
2	20.942	11764780	1.05	linalool	92
3	23.655	29498356	2.63	methyl chavicol	95
4	24.945	33574388	2.99	d-carvone	87
5	25.232	5075938	0.45	N-(4-benzyloxybenzyl) 4-methoxybenzenamine	86
6	25.831	970600193	86.40	trans-anethole	98
7	31.269	8778123	0.78	benzocycloheptene	98
8	32.035	42582400	3.79	α-gurjunene	94

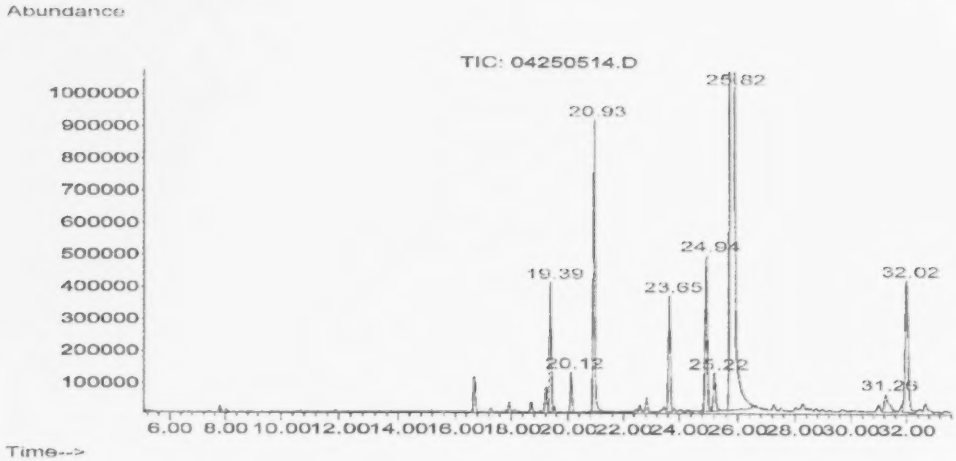
ANISE #1



ANISE # 1

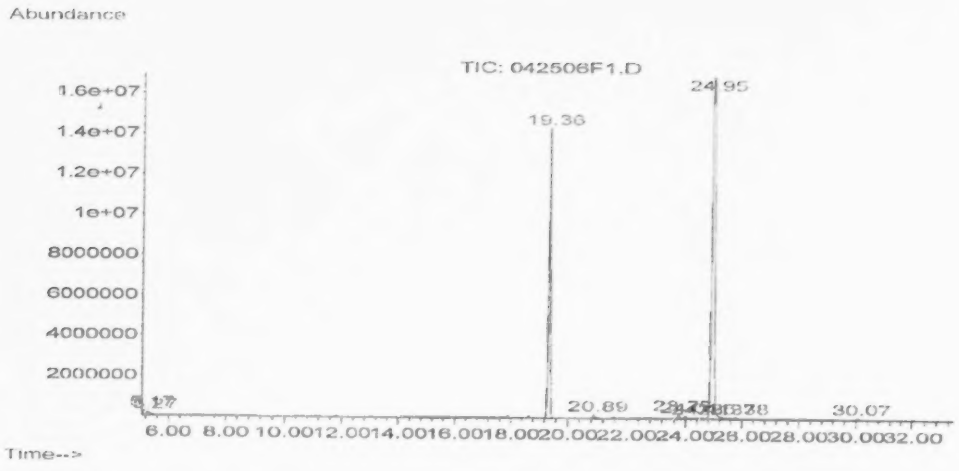
Peak#	Ret Time	Area	% Area	Identification	% Prob. Of ID
1	19.389	15232623	1.45	limonene	94
2	20.936	7617040	0.73	linalool	92
3	23.651	20494795	1.95	methyl chavicol	95
4	24.940	23558786	2.24	d-carvone	87
5	25.826	940924550	89.57	trans-anethole	98
6	26.071	7240561	0.69	cis-anethole	91
7	31.255	5187388	0.49	benzocycloheptene	98
8	32.025	30215621	2.88	α-gurjunene	94

ANISE/CORIANDER #1



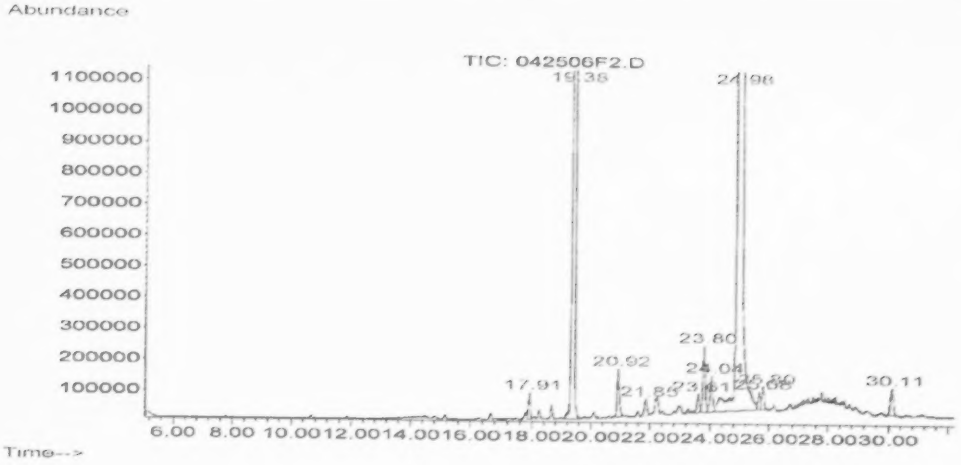
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	19.386	17238888	1.66	limonene	95
2	20.123	5417561	0.52	γ-terpinene	86
3	20.933	40408144	3.89	linalool	92
4	23.647	16838264	1.62	methyl chavicol	94
5	24.939	25623795	2.47	d-carvone	90
6	25.217	6356044	0.61	unknown	
7	25.820	888620950	85.51	trans-anethole	97
8	31.260	6801782	0.65	1H-bezocycloheptene	97
9	32.017	31908466	3.07	α-gurjunene	94

CARAWAY #1



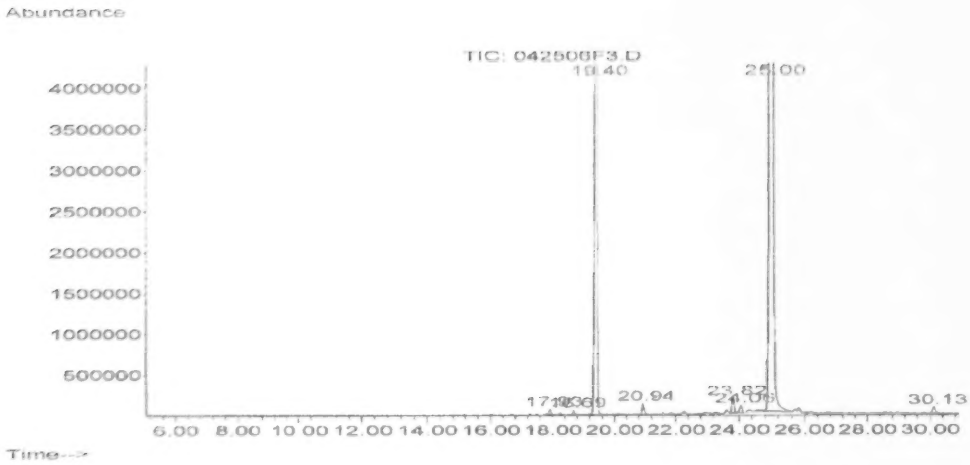
Peak#	Ret Time	Area	% Area	Identification	% Prob. Of ID
1	19.355	594123117	29.54	limonene	94
2	24.947	1342724530	66.77	carvone	95

CARAWAY #2



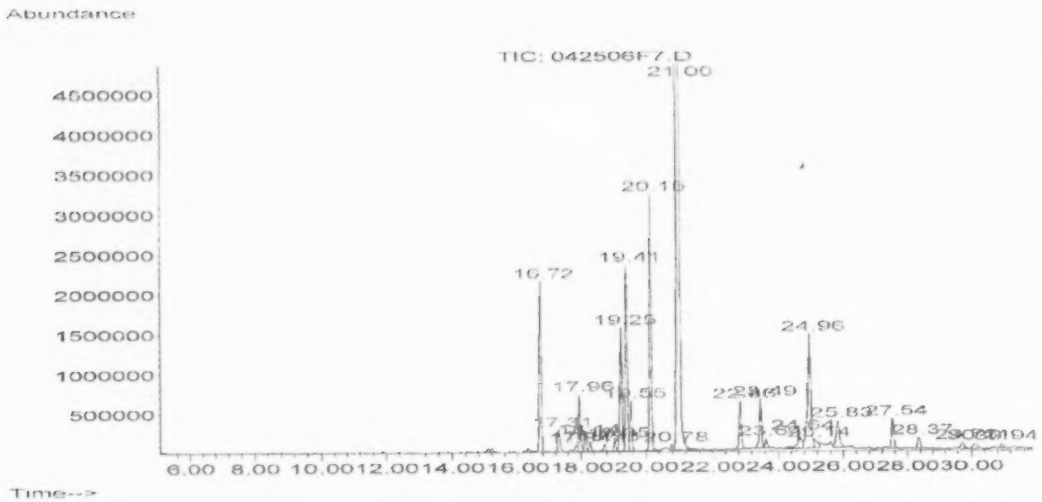
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	17.908	3686326	0.20	β-pinene	90
2	19.380	456185362	25.02	limonene	94
3	20.918	7914332	0.43	linalool	76
4	21.849	3340522	0.18	unknown	
5	23.608	4206437	0.23	α-terpineol	60
6	23.799	11389915	0.62	unknown	
7	24.043	6944946	0.38	unknown	
8	24.976	1316703320	72.23	carvone	92
9	25.680	3176388	0.17	unknown	
10	25.803	4094958	0.22	unknown	
11	30.107	5401962	0.30	caryophyllene	89

CARAWAY #3



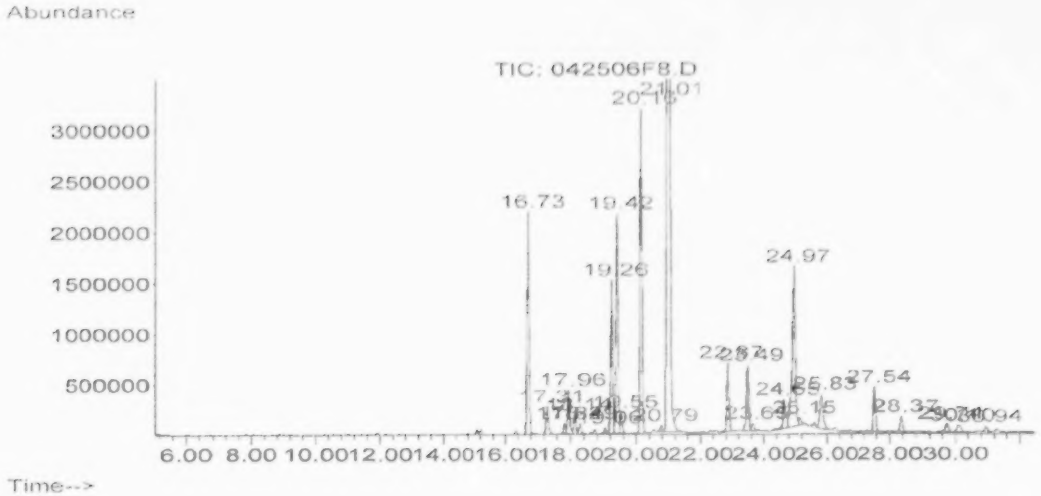
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	19.400	453783305	26.80	limonene	96
2	24.999	1209239173	71.41	carvone	94
3	30.134	4925742	0.29	caryophyllene	95

CORIANDER #1



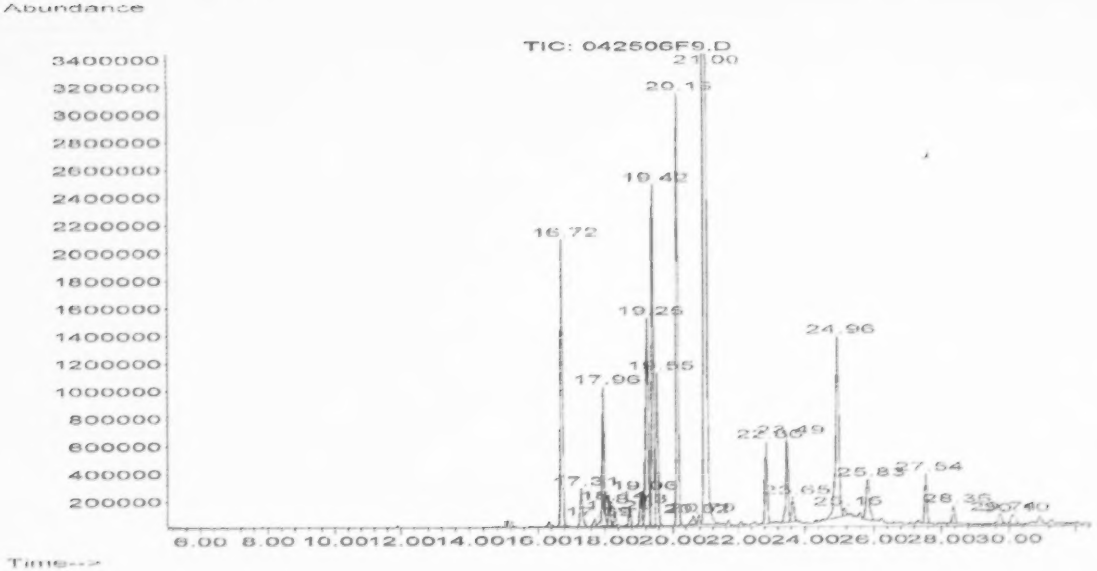
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.722	92620094	5.97	α-pinene	94
2	17.306	13457810	0.87	camphene	91
3	17.842	3360799	0.22	β-phellandrene	72
4	17.956	30342536	1.96	β-pinene	78
5	18.139	8561999	0.55	pinene	94
6	18.288	4616613	0.30	octanal	93
7	18.728	4674737	0.30	unknown	
8	19.052	6245160	0.40	α-terpinene	91
9	19.253	75388273	4.86	*o*-cymene	90
10	19.414	98053399	6.32	limonene	87
11	19.547	25971454	1.67	3-carene	91
12	20.152	137176704	8.84	β-terpinene	80
13	20.783	3015647	0.19	2-hexyl-furan	78
14	21.000	792925541	51.09	linalool	93
15	22.859	30206852	1.95	l-camphor	91
16	23.487	28675257	1.85	decanal	92
17	23.644	5879510	0.38	*p*-menth-1-en-8-ol	91
18	24.640	16022881	1.03	(E)-3,7-dimethyl-2,6-octadien-1-ol	80
19	24.964	98394181	6.34	carvone	64
20	25.136	8002992	0.52	cis-3-methyl-2- pentene	50
21	25.833	25672960	1.65	*p*-anethole	93
22	27.540	19279809	1.24	geranyl acetate	90
23	28.365	7727309	0.50	dodecanal	60
24	29.734	5641868	0.36	unknown	
25	30.107	6845431	0.44	1-tetradecene	47
26	30.938	3138501	0.20	unknown	

CORIANDER #2



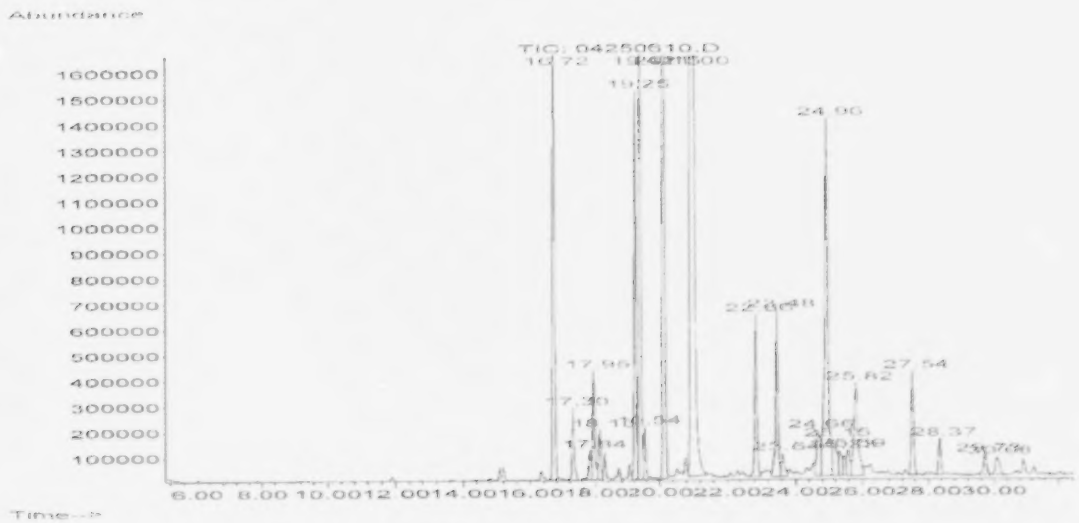
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.728	94418697	5.93	α-pinene	94
2	17.311	12907483	0.81	camphene	91
3	17.845	4743080	0.30	β-pellandrene	72
4	17.961	19121205	1.20	β-pinene	78
5	18.143	8737839	0.55	pinene	84
6	18.292	4755886	0.30	octanal	93
7	19.058	3097203	0.19	α-terpinene	91
8	19.259	68195259	4.28	*o*-cymene	90
9	19.418	92302388	5.79	limonene	87
10	19.549	9012757	0.57	3-carene	91
11	20.156	137049337	8.60	β-terpinene	80
12	20.789	2459127	0.15	2-hexyl-furan	78
13	21.009	876490382	55.02	linalool	93
14	22.865	30908627	1.94	l-camphor	91
15	23.491	30967561	1.94	decanal	92
16	23.653	3914488	0.25	*p*-menth-1-en-8-ol	91
17	24.647	19070227	1.20	(E)-3,7-dimethyl-2,6-octadien-1-ol	80
18	24.970	99573061	6.25	carvone	90
19	25.154	3866194	0.24	cis-3-methyl-2-pentene	62
20	25.834	24736607	1.55	*p*-anethole	90
21	27.544	22271724	1.40	geranyl acetate	90
22	28.371	8088271	0.51	dodecanal	70
23	29.742	6010152	0.38	unknown	
24	30.101	6880324	0.43	1-tetradecene	47
25	30.940	3495415	0.22	unknown	

CORIANDER #3



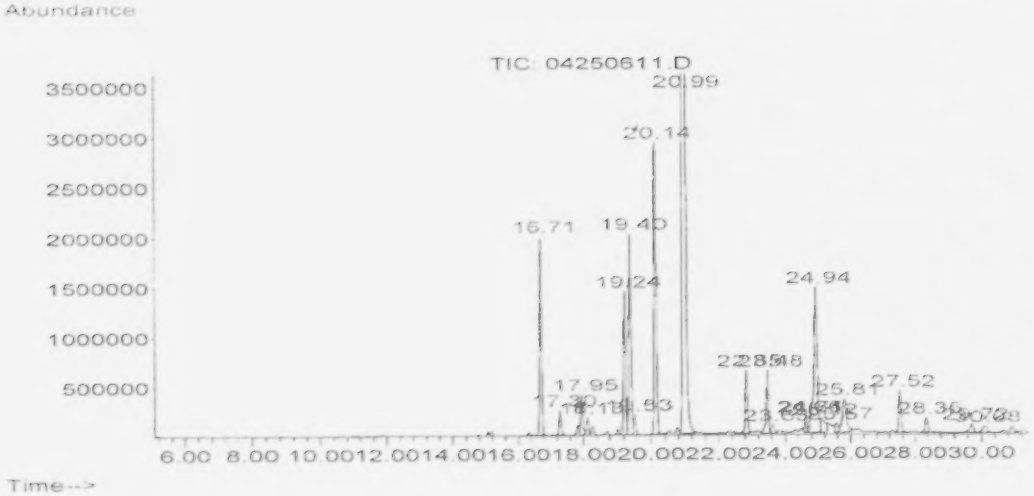
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.725	88578127	6.19	α-pinene	94
2	17.308	13459608	0.94	camphene	91
3	17.693	2545245	0.18	β-phellandrene	72
4	17.958	42832345	2.99	β-pinene	78
5	18.141	7446780	0.52	pinene	94
6	18.290	3633817	0.25	octanal	93
7	18.733	6937999	0.48	α-thujene	72
8	19.055	9721602	0.68	α-terpinene	91
9	19.254	80287599	5.61	*o*-cymene	90
10	19.417	104828942	7.32	limonene	87
11	19.548	45948623	3.21	3-carene	91
12	20.153	131795099	9.21	β-terpinene	80
13	20.617	4232672	0.30	unknown	
14	20.787	2867947	0.20	2-hexyl-furan	78
15	20.997	674596016	47.14	linalool	93
16	22.858	28403808	1.98	l-camphor	91
17	23.487	28351131	1.98	decanal	92
18	23.648	9181175	0.64	α-terpineol	91
19	24.963	84321253	5.89	carvone	64
20	25.151	2735725	0.19	cis-3-methyl-2-pentene	49
21	25.828	21735995	1.52	*p*-anethole	90
22	27.543	17662483	1.23	geranyl acetate	90
23	28.365	7053708	0.49	dodecanal	91
24	29.739	5305050	0.37	unknown	
25	30.099	6673192	0.47	1-tetradecene	52

CORIANDER #4



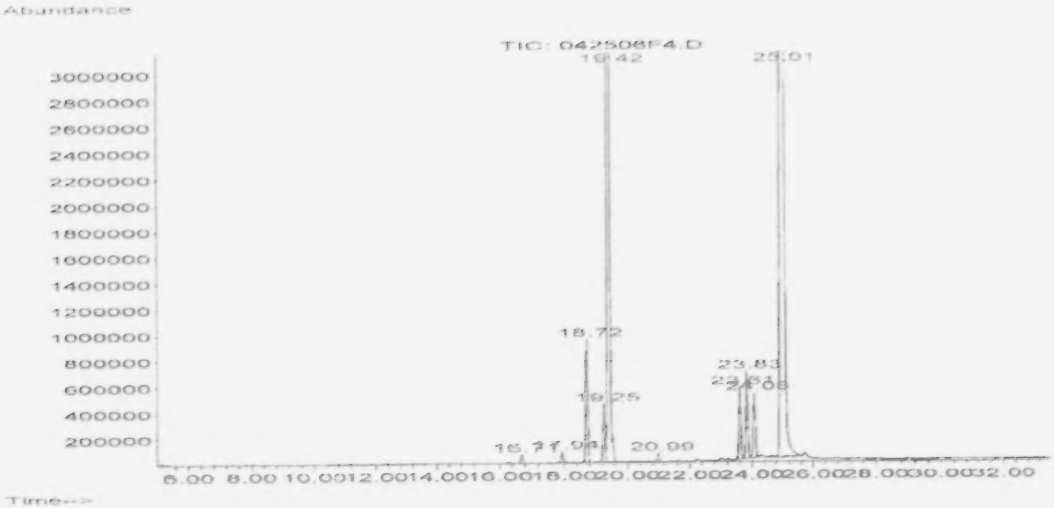
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.719	93448944	6.04	α-pinene	91
2	17.302	13002894	0.84	camphene	94
3	17.840	5207149	0.34	β-phellandrene	78
4	17.951	18282147	1.18	β-pinene	91
5	18.135	8672376	0.56	pinene	94
6	19.251	66993430	4.33	*o*-cymene	90
7	19.410	90203165	5.83	limonene	94
8	19.542	8712478	0.56	3-carene	91
9	20.147	132339538	8.56	β-terpinene	80
10	20.999	831966208	53.79	linalool	93
11	22.855	30510957	1.97	l-camphor	91
12	23.481	31420938	2.03	decanal	92
13	23.640	5549937	0.36	4-carene	55
14	24.664	13182950	0.85	(E)-3,7-dimethyl-2,6-octadien-1-ol	80
15	24.959	100243859	6.48	carvone	90
16	25.147	11743565	0.76	cis-3-methyl-2-pentene	62
17	25.327	5396862	0.35	9-tetradecenal	86
18	25.589	6827741	0.44	unknown	
19	25.824	32079473	2.07	2-carboxycinnamic acid	47
20	27.536	20246680	1.31	geranyl acetate	91
21	28.368	7727040	0.50	2-decen-1-ol	52
22	29.730	5915199	0.38	unknown	
23	30.084	7053231	0.46	(E)-2-tridecenal	55

CORIANDER #5



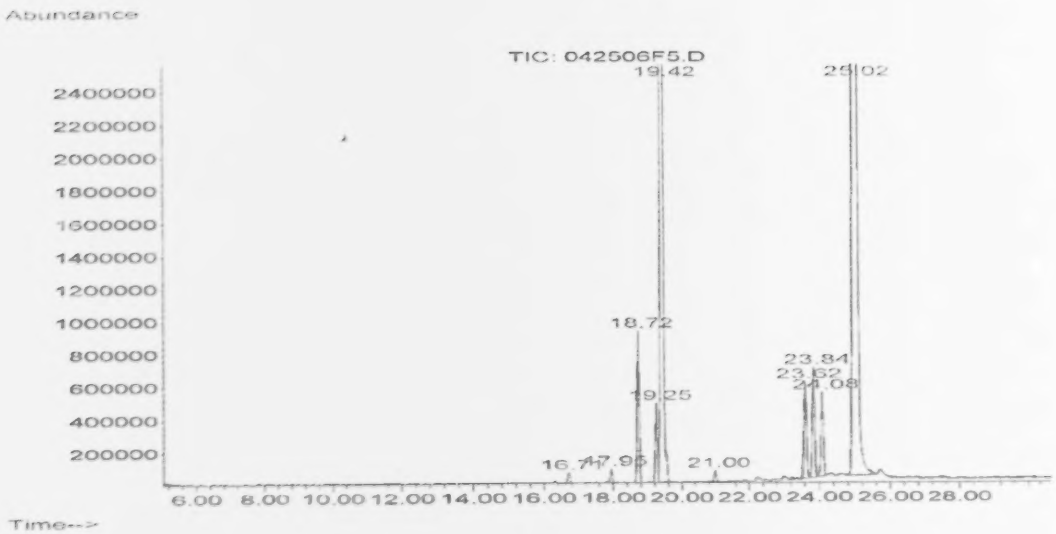
Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.712	86130992	5.80	α-pinene	94
2	17.297	12063329	0.81	camphene	91
3	17.947	17362516	1.17	β-myrcene	86
4	18.126	7793594	0.52	α-pinene	95
5	19.245	63211960	4.25	*o*-cymene	90
6	19.403	85855153	5.78	limonene	90
7	19.535	8493317	0.57	3-carene	91
8	20.141	124696848	8.39	β-terpinene	80
9	20.990	809331928	54.46	linalool	93
10	22.846	30419320	2.05	l-camphor	91
11	23.477	30198322	2.03	decanal	92
12	23.634	5176410	0.35	methylethyl cyclohexene	92
13	24.656	7977642	0.54	(E)-3,7-dimethyl-2,6-octadien-1-ol	81
14	24.708	5172499	0.35	(Z)-3,7-dimethyl-2,6-octadien-1-ol	86
15	24.944	101763706	6.85	carvone	76
16	25.120	12816765	0.86	unknown	
17	25.571	5655030	0.38	2-n-octylfuran	58
18	25.814	29733606	2.00	2-carboxycinnamic acid	47
19	27.524	21325794	1.44	geranyl acetate	91
20	28.349	7989479	0.54	1,1-dodecanediol	74
21	29.718	6180509	0.42	unknown	
22	30.079	6652088	0.45	caryophyllene	53

DILL #1



Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.708	2767581	0.15	α-pinene	96
2	17.943	3692976	0.20	β-pinene	91
3	18.718	41709295	2.21	α-thujene	80
4	19.248	19930973	1.05	*o*-cymene	91
5	19.418	550262078	29.11	limonene	97
6	20.992	3086459	0.16	*p*-cymenene	90
7	23.610	25833310	1.37	unknown	
8	23.835	31051350	1.64	unknown	
9	24.078	24254740	1.28	d-dihydrocarvone	95
10	25.012	1187946715	62.84	carvone	94

DILL #2



Peak#	Ret Time	Area	% Area	Identification	% Prob. of ID
1	16.712	2753296	0.14	α-pinene	96
2	17.950	3988065	0.20	β-pinene	91
3	18.724	40798156	2.08	α-thujene	80
4	19.254	21498598	1.10	*o*-cymene	91
5	19.425	576784193	29.44	limonene	97
6	20.995	3879881	0.20	*p*-cymenene	90
7	23.615	27662078	1.41	unknown	
8	23.841	30997582	1.58	unknown	
9	24.083	24778285	1.26	d-dihydrocarvone	95
10	25.017	1226307336	62.58	carvone	94

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